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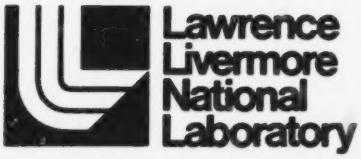
Lower-Bound K_{Iscc} Values for Bolting Materials - A Literature Study

Alfred Goldberg and Mary C. Juhas

Prepared for
U.S. Nuclear Regulatory Commission



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Lower-Bound K_{Iscc} Values for Bolting Materials - A Literature Study

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ABSTRACT

An extensive literature survey on stress-corrosion cracking of a variety of steels was made in response to the need by the U.S. Nuclear Regulatory Commission (NRC) to establish lower-bound K_{Iscc} values for bolting type materials. Materials evaluated include the heat-treatable plain-carbon and Cr-Mo and Cr-Mo-Ni low-alloy steels, 17-4 PH and Custom 455 precipitation-hardening stainless steels, and the 18Ni-maraging steels. An exhaustive survey was made for water, aqueous chloride, and aqueous sulfide environments. We also report limited data on H_2S and H_2 gases, aqueous H_3BO_4, and atmospheric environments. The data are presented in the form of K_{Iscc} versus yield strength. The proposed NRC lower-bound K_{Iscc} curve for the low-alloy steels is consistent with the data reported for the various aqueous environments, but excluding the sulfides. The corresponding lower-bound curve based on reported data for 18Ni-maraging steels falls below that proposed by the NRC, especially at the high strength levels. The lower-bound curves for the precipitation-hardening stainless steels fall below the lower-bound curve for the maraging steels at the lower strength levels, but they merge at a yield strength of approximately 220 ksi. Reference is also made to crack-growth rate (CGR) and time to failure (t_f). These two parameters frequently were found to vary differently from the corresponding K_{Iscc} values when examined as a function of material or environmental variables. The influence of various material and environmental factors on K_{Iscc}, CGR, and t_f are discussed.

EXECUTIVE SUMMARY

The U.S. Nuclear Regulatory Commission (NRC) staff, in charge of Generic Activity A-12, is establishing acceptance criteria for the structural integrity of support members for a number of nuclear-reactor plant components. Included in this task is the evaluation of high-strength bolting materials using a fracture mechanics approach. Because of a history of bolting failures by stress corrosion cracking (SCC) reliable lower-bound K_{Iscc} (threshold stress intensity for SCC) values had to be established. To obtain the necessary data we made a literature survey of SCC of bolting-type materials.

Materials evaluated include the heat-treatable plain-carbon and Cr-Mo, and Cr-Mo-Ni low-alloy steels, 17-4 PH and Custom 455 stainless steels, and the 18Ni-maraging steels. The environments for which an exhaustive study was made are water, aqueous chlorides, aqueous sulfides, and to a limited extent are H_2S and H_2 gases, aqueous H_3BO_4, and atmospheric. The data are presented in the form of K_{Iscc} versus σ_y (yield strength).

Considerable scatter in the plotted data is evident for virtually all the alloys examined. This is attributed to a combination of factors possibly involving variations in melting practice, heat treatment, microstructure, and composition. Differences in testing procedures, especially exposure periods, may also have contributed to such scatter. Little or no difference exists in K_{Iscc} between distilled H_2O and various aqueous chlorides (primarily NaCl); the K_{Iscc} is essentially constant over a pH range from 2 to 9. The pH as well as large differences in H_2S content will affect the K_{Iscc} values in aqueous sulfides. The effects of material variables and environmental variables on K_{Iscc}, crack-growth rate (CGR) and time to failure (t_f) are discussed. The effects of material variables are often controversial, while the effects of environmental variables are relatively self-consistent. A number of examples are presented where some variable may have little or no effect on K_{Iscc} but may result in a large increase in CGR and decrease in t_f. We would therefore caution the use of K_{Iscc} exclusive

to CGR and t_f; for example, K_{Iscc} may be based on tests with insufficient exposure time; the material may have been improperly heat treated; stray voltages may result in polarization potentials; transients may result in stress excursions developing stress intensities exceeding K_{Iscc}.

The lower-bound curves for water and the various aqueous chloride environments exhibit a sharp drop in K_{Iscc} with an increase in σ_y. For the low-alloy steels above about 190 ksi the lower-bound values of K_{Iscc} become σ_y-insensitive at about 10 ksi·in.$^{1/2}$. For the highly alloyed steels the σ_y at which K_{Iscc} becomes insensitive is increased to higher values. For maraging steels this occurs at about 240 ksi σ_y with the lower-bound K_{Iscc} close to 7 ksi·in.$^{1/2}$.

The lower-bound K_{Iscc} curve proposed by the NRC staff for low-alloy steels is consistent with the data obtained for water and various aqueous chloride environments. That proposed by the NRC for the maraging steel is non-conservative, especially at the high σ_y levels; at 240 ksi it is higher by about 25 ksi·in.$^{1/2}$ than that based on the surveyed data. At the lower σ_y levels, below about 220 ksi, the lower-bound curve for maraging steels lies significantly above that for the low-alloy steels. The curves obtained for two precipitation-hardening stainless steels fall just below the curve for the maraging steels up to σ_y of about 220 ksi; the three curves appear to merge at stresses above this value.

The presence of environments, other than aqueous chlorides and water, may require a significant drop in the proposed lower-bound K_{Iscc} values, as manifested by the plots for the sulfide environments. At σ_y of 120 ksi, a reference value in the NRC evaluation, the presence of aqueous H_2S can cause a drop in K_{Iscc} by some 50 ksi·in.$^{1/2}$ relative to K_{Iscc} for an aqueous chloride environment. Thus, a record of the environmental history during the operation of the nuclear-reactor plant could be a critical component in the final assessment of the bolting problem. We did not evaluate data on corrosion-assisted fatigue, and this failure mechanism, where operative, must also be factored into the analysis.

CONTENTS

Acknowledgments iii

Abstract v

Executive Summary vii

List of Illustrations x

1. Introduction 1

2. Scope 2

   2.1 Data Acceptance Criteria 3

   2.2 Materials 4

   2.3 Material and Processing Variables 5

   2.4 Environmental Variables 5

3. K_{Iscc} Versus σ_y Plots 7

   3.1 SAE 4340 Steels 8

   3.2 SAE 43XX-Type of Steels 12

   3.3 SAE 41XX-Type of Steels 12

   3.4 D6AC Steel 18

   3.5 H-11 Die Steel 18

   3.6 HY Steels 18

   3.7 Miscellaneous Low-Alloy Steels 22

   3.8 Precipitation Hardening Stainless Steels 22

   3.9 18Ni-Maraging Steels 28

4. Discussion on Influencing Factors 28

   4.1 Compositional Effects 31

   4.2 Microstructural Effects 34

   4.3 Prestressing and Prior Deformation Effects 36

   4.4 Environmental Effects 36

5. Summary 41

6. References 43

LIST OF ILLUSTRATIONS

1. K_{Iscc} versus yield strength for SAE 4340 steel reported for "combined" aqueous data 9

2. K_{Iscc} versus yield strength for SAE 4340 steel reported for various environments containing water or aqueous chlorides 10

3. K_{Iscc} and K_{IH_2S} versus yield strength for SAE 4340 steel reported for various H_2S environments and for H_3BO_4 solution 11

4. K_{Iscc} versus yield strength for several high-strength heat-treated SAE 43XX-type steels reported for various types of environments . . . 13

5. K_{IH_2} versus yield strength for SAE 4335V, SAE 4340, and 300M steels reported for H_2 gas 14

6. K_{Iscc} versus yield strength for SAE 4130 and SAE 4140 steels reported for "combined," aqueous NaCl, and distilled H_2O environments 15

7. K_{Iscc} versus yield strength for several SAE 41XX-type steels reported for H_2S aqueous environments 16

8. K_{IH_2} versus yield strength for several SAE 41XX steels reported for H_2 gas 17

9. K_{Iscc} versus yield strength for D6AC steel reported for "combined," aqueous NaCl, and distilled H_2O environments 19

10. K_{Iscc} versus yield strength for H-11 die steel reported for various environments containing water or aqueous chlorides 20

11. K_{Iscc} versus yield strength for HY steels reported for various environments containing water, aqueous chlorides, and/or H_2S 21

12. K_{Iscc} versus yield strength for miscellaneous low-alloy steels reported for "combined" environments 23

13. K_{Iscc} versus yield strength for miscellaneous low-alloy steels reported for H_2S environments 24

14. K_{IH_2} versus yield strength for several different types of steels reported for H_2 gas 25

15. K_{Iscc} or K_{IH_2S} versus yield strength for Custom 455 stainless steel reported for various environments containing water, aqueous chlorides, air, H_2S gas, or H_3BO_4 solution 26

16. K_{Iscc} versus yield strength for 17-4 PH stainless steel reported for "combined" or specific aqueous chloride environments . . 27

17. K_{Iscc} versus yield strength for 18Ni-maraging steels reported for "combined" aqueous environments 29

18. K_{Iscc} versus yield strength for 18Ni-maraging steels reported for water, air, and various aqueous chloride environments 30

19. Effect of various impurities and alloying elements on K_{Iscc} for ASE 43XX-type steels.[25] 33

LOWER BOUND K_{Iscc} VALUES FOR BOLTING MATERIALS — A LITERATURE SURVEY

Alfred Goldberg and Mary C. Juhas

1. INTRODUCTION

A literature survey was made for the U.S. Nuclear Regulatory Commission (NRC) to determine lower-bound values of K_{Iscc} (threshold stress intensity for stress-corrosion cracking below which crack growth does not occur). The materials evaluated were bolting-type steels exposed to environments that might be related to operating conditions in nuclear-reactor plants. At the time this study was initiated, the NRC staff was concerned with formulating the final guidelines of acceptance criteria that would assure the structural integrity of support members for a number of nuclear-reactor plant components. The concerns and preliminary guidelines were documented in detail by NRC staff responsible for Generic Activity A-12.[1-3] One of these guidelines addressed the problem related to evaluating the integrity of high-strength bolting materials using a fracture mechanics approach. In particular, there was a need to develop a reliable lower-bound K_{Iscc} base that would permit establishing conservative and meaningful allowable loading limits for stressed bolts.

The bolting materials used in the reactor plants,[1,2] include moderate-, high-, and ultra-high-strength steels consisting of plain-carbon steels, Cr-Mo and Cr-Mo-Ni low-alloy steels, 17-4 PH and Custom 455 stainless steels, and 18Ni-maraging steels. In many cases, the ASTM specifications allow a wide range of compositions, with usually only the lower limit of tensile properties being designated. Thus, some of these

bolts may have much higher yield strengths (σ_y) than are normally expected. It is well known that these steels, when heat-treated to high strength levels, are likely to be highly susceptible to stress-corrosion cracking (SCC). A sharp drop in K_{Iscc} occurs at some moderately high stress level depending on the class of alloys and the environment.[4-6] Thus, the use of steels at excessively high-strength levels may lead to early failure.

There is also a significant decrease in K_{Ic} with increasing σ_y; however, it is not as dramatic as the corresponding decrease in K_{Iscc}. To determine a safe upper-limit for σ_y where SCC might occur, it is therefore especially important to establish the position of the K_{Iscc} lower-bound values in the region of rapid drop-off. Although there is a general decrease in K_{Iscc} with a decrease in K_{Ic}, correlations between these two parameters for a given alloy appear to be poor when related to variations in alloy composition and thermomechanical history. Evidence of opposing trends will be presented later in the report. Thus, it is important to recognize the danger of using estimated K_{Iscc} values based on combining limited K_{Iscc} with K_{Ic} data. The need for actual K_{Iscc} data for various alloy histories and compositions is also addressed in this report.

Documentation exists of a number of bolting failures in nuclear reactor plants. Although no special effort was made to review the history of such failures, some of the nuclear-related papers on bolting failures are referenced here.[7-10]

2. SCOPE

The scope of this study consisted of the following tasks:

1. To perform a literature survey and analysis on SCC of bolting-type materials in various environments that possibly reflect conditions at support structures in reactor plants (e.g., PWR steam generator and coolant pump supports).[1,2]

2. To establish lower-bound K_{Iscc} versus σ_y limits for different classes of materials.

3. To examine the effects of material variables, such as impurities, alloying composition, heat treatment, microstructure, etc., on K_{Iscc}.

4. To examine the effects of environmental variables such as relative humidity (RH), pH, concentration, temperature, polarization potential, etc, on K_{Iscc}.

2.1 DATA ACCEPTANCE CRITERIA

The survey was restricted largely to σ_y versus K_{Iscc} information. Where σ_y was not given, it was estimated from the reported hardness or tensile strength; these data are identified as such. The validity of K_{Ic} data are met when the following equations are simultaneously satisfied:

$$a_{min} = B_{min} = (W-a)_{min} = \frac{W_{min}}{2} = 2.5 \left(\frac{K_{Iscc}}{\sigma_y} \right)^2 \qquad (1)$$

where a_{min}, B_{min}, and W_{min} are the minimum crack length, minimum specimen thickness, and minimum specimen width, respectively. As yet, there are no established ASTM requirements for K_{Iscc}. The practice has been to follow the requirements for K_{Ic}, however, the coefficient 2.5 may be too conservative.

A number of different specimen configurations and test procedures were reported. Constant-load specimens lead to increasing K_I values with crack growth. Here, the determination of K_{Iscc} generally involved either continuous or, more frequently, incremental load increases until

crack growth was observed. This point was usually, but not always, verified by loading a new specimen just below this threshold stress intensity for some extended length of time. Constant deflection tests lead to decreasing K_I values as the crack propagates. Typically, a number of specimens were tested with different initial stress intensities (K_{Ii}); K_{Iscc} corresponds then to the value of K_{Ii} that would not lead to any crack growth in some specified time. Frequently, specimen dimensions were not indicated; however, the claim was usually made that these were within ASTM specifications for valid K_{Ic} determinations.

A possible equal or even more serious consideration than the presence of plane-strain conditions is the extensive range in testing times reported by different investigators; in a number of cases these times were not clearly indicated. Examples of the importance of having exposure times ranging into many thousands of hours to provide reliable K_{Iscc} values will be given later. Crack blunting caused by excessive K values in fatigue precracking (stress ranges often are not given) and during actual testing, especially in the variable constant-load tests, may affect the observed K_{Iscc} value. Because of these problems, all of which will usually lead to non-conservative rather than conservative K_{Iscc} values, we disregarded validating the reported data by use of Eq. 1. Departure from plane-strain conditions should also lead to non-conservative results. Although the use of invalid data would tend to increase scatter, such data should not fall below the lower-bound curves established for "valid" data.

2.2 MATERIALS

Bolting materials used in nuclear-reactor plants are mostly of compositions comparable to AISI 4140 and 4340.[1,2] Fortunately, much of the work surveyed covered these two classes of alloys and they were evaluated as separate groups. All remaining low-alloy and plain-carbon steels were combined as a third group. While H-11, 17-4 PH, Custom 455, and 18Ni-maraging steels were treated as four distinct groups. Data from experimental alloys with compositions departing from commercial alloys are identified as such.

2.3 MATERIAL AND PROCESSING VARIABLES

Alloy content, impurity content, melting and processing variables, heat-treatment, and microstructure all affect K_{Ic} values. Much work has been reported on attempts to determine the effect of these parameters on K_{Iscc}. While large improvements in K_{Ic} over a wide range of σ_y have resulted from microstructural refinement, melting practice, and impurity control, the effect of such material variables on K_{Iscc} usually appear to be minor, especially in the higher σ_y range. In the lower σ_y range (>150 ksi) however, the quenched-and-tempered structures are generally more resistant to SCC than are the normalized-and-tempered or bainitic-and-tempered structures of equal strengths. Because of a primary interest in the high strength range (≥150 ksi) the steels were not separated according to melting practice, minor differences in composition, or heat treatment. However, examples will be presented where material parameters do or do not show any effects. Finally, it was noted that differences in results reported by different investigators on apparently the same materials were usually greater than variations in results reported for a specific study concerned with the effects of material parameters.

2.4 ENVIRONMENTAL VARIABLES

There is considerable evidence in the literature that supports the contention that hydrogen attack,[11-13] as well as anodic dissolution, contributes to SCC in aqueous solutions and, in fact, these two mechanisms may act conjointly or successively in a given environment.[14] Although the rate-controlling process may be different in various hydrogen media (molecular, atomic, precharged, aqueous), thus affecting the crack-growth rate (CGR) in stage II, the K_{Iscc} or K_{IH_2}* does not vary significantly with the source of hydrogen.

---

* The term K_{IH_2} is used here for the threshold stress in hydrogen gas or hydrogen-charged environments.

Therefore, data on hydrogen studies have been included in the analysis. Some recent work in France[15] on several low-alloy steels tested in gaseous hydrogen and in aqueous NaCl suggests that $K_{IH_2} > K_{Iscc}$; however, the time to failure (t_f) above the threshold value is considerably less in H_2 than it is in the aqueous environment. The ranking of the various steels examined were the same for the two environments.[15] The data plotted by Sandoz for 4340 steels are in general agreement with these results in that $K_{IH_2} > K_{Iscc}$ (salt water) $> K_{IH}$ (cathodic charge).[12]

Since exposures to coastal and industrial environments are likely to have occurred, data has been included for salt water (NaCl solutions, sea water, sea coasts, and artificial sea water) and sulfide (H_2S aqueous and H_2S gas) environments. The data show that K_{Iscc} decreases with increase in humidity and that there is little difference between distilled water and NaCl solutions. However, sea-water environments are generally more damaging than are the NaCl solutions. The rapid drop in K_{Iscc} with an increase in σ_y occurs at much lower stresses in sulfide than in salt solutions. Thus, a lower-bound curve based on sulfide solutions is probably over-conservative for reactor plant environments; however, these data do point to the dangers of industrial sulfide-bearing environments. Exact compositions of the various environments are not specifically identified in our paper. The NaCl solutions range from approximately 2 to 5 wt. %; the sulfide solutions range from H_2S in distilled water to the NACE solution (5% NaCl + 0.5% acetic acid, saturated with H_2S).

For water and aqueous chlorides there is little, if any, effect of pH on K_{Iscc} over the range of pH about 2 to 9.[4] This is consistent with the observation that the pH and solution potential at the crack tip are virtually independent of the conditions external to the crack tip.[13,16] Thus, the pH was not considered as a variable for these environments. For aqueous sulfides the susceptibility to cracking does increase with pH over this same range. Nevertheless, the possible effects of pH were not isolated.

There is a minor effect of polarization potential on K_{Iscc}. However, the corresponding effect on CGR can be significant; the minimum rate occurs near the open-circuit potential. Either high cathodic (atomic hydrogen) or anodic (molecular hydrogen and/or anodic dissolution) reactions can result in an increase of several orders of magnitude in CGR. The selection of data was restricted to freely corroding potential tests. If applied potentials are used in the reactor plants to reduce corrosion (or if stray voltages exist), the lower-bound values for freely corroding conditions are likely to be too conservative. Some examples of the influence of polarization will be presented.

Data of K_{Iscc} versus σ_y are presented to provide a guide for establishing lower-bound curves for the alloys and environments outlined in the above. The lower-bound curves originally proposed by the NRC are included in all figures. The presentation of data will be followed with a detailed discussion on the effects of various material and environmental variables.

3. K_{Iscc} VERSUS σ_y PLOTS

The data are categorized according to type of steel and further subdivided according to test environment. Some of the data were extracted from review articles where different environments having similar effects had been grouped together. Identification of the specific environment for each of these data points was not always possible as some of the original references were not available. Therefore, those environments that include various purities of H_2O, aqueous NaCl, simulated sea water, sea water, and coastal environments were plotted together as "combined." Variations in K_{Iscc} resulting from testing of high-strength steels in these different aqueous environments usually can be neglected.[4,6,17] Some of the data in the combined plots are likely to be repeated in plots for the individual environments. Duplicate values were omitted in plots that contain a high

density of data. All available SCC data for the aqueous (H_2O, NaCl, sea waters, and H_2S) environments were included. Data for gaseous (H_2 and H_2S) environments were obtained only incidental to the search for the aqueous environments. The role of gaseous-induced cracking is likely to be secondary to that caused by the aqueous environments.

3.1 SAE 4340 STEELS:

Figure 1 contains the "combined" aqueous data for SAE 4340 steels.[4,5,17-46] The solid line, A-A, in all figures refers to the lower-bound K_{Iscc} curve for low-alloy steels originally specified by Task Group A-12.[1-3] Figure 2 contains 4340 data separated according to specific aqueous environments.[4,5,12,18,21-23,25,27-29,31,38,40,41,43,44,46-60] Data for 4340 exposed to H_2S environments are plotted in Fig. 3 for dry gas,[4,49,55,61-63] wet gas,[64,65] and aqueous solutions.[4,39,66-68] For the aqueous H_2S solutions, no distinction was made between those containing only H_2S and those containing additions of chlorides and acetic acid. There does not appear to be any significant effect of chloride additions on the K_{Iscc}, although the acidified solutions accelerate the cracking process.[66] Too low an H_2S concentration in aqueous solutions may result in excessive K_{Iscc} values.[66] The H_2S gas was assumed to be dry unless specified otherwise.

Based on the water and aqueous chlorides data, in Figs. 1 and 2, the proposed NRC lower-bound curve, A-A, is reasonable and not over-conservative. We believe that the large scatter in data for a given σ_y is most likely due to variations in material composition, manufacturing history, and heat treatment, although the role of these variables is not too clear. It is very likely that some of the scatter is also due to variations in testing methods, exposure times, and interpretation of test results. Above about 190 ksi, σ_y has little affect on K_{Iscc}. Below this value K_{Iscc} rises rapidly with a decrease in σ_y.

As can be seen from Fig. 3 the presence of H_2S, especially when associated with H_2O has a large effect on lowering K_{Iscc} over a wide range of σ_y. The lower-bound curve for the plotted data is given by curve B-B.

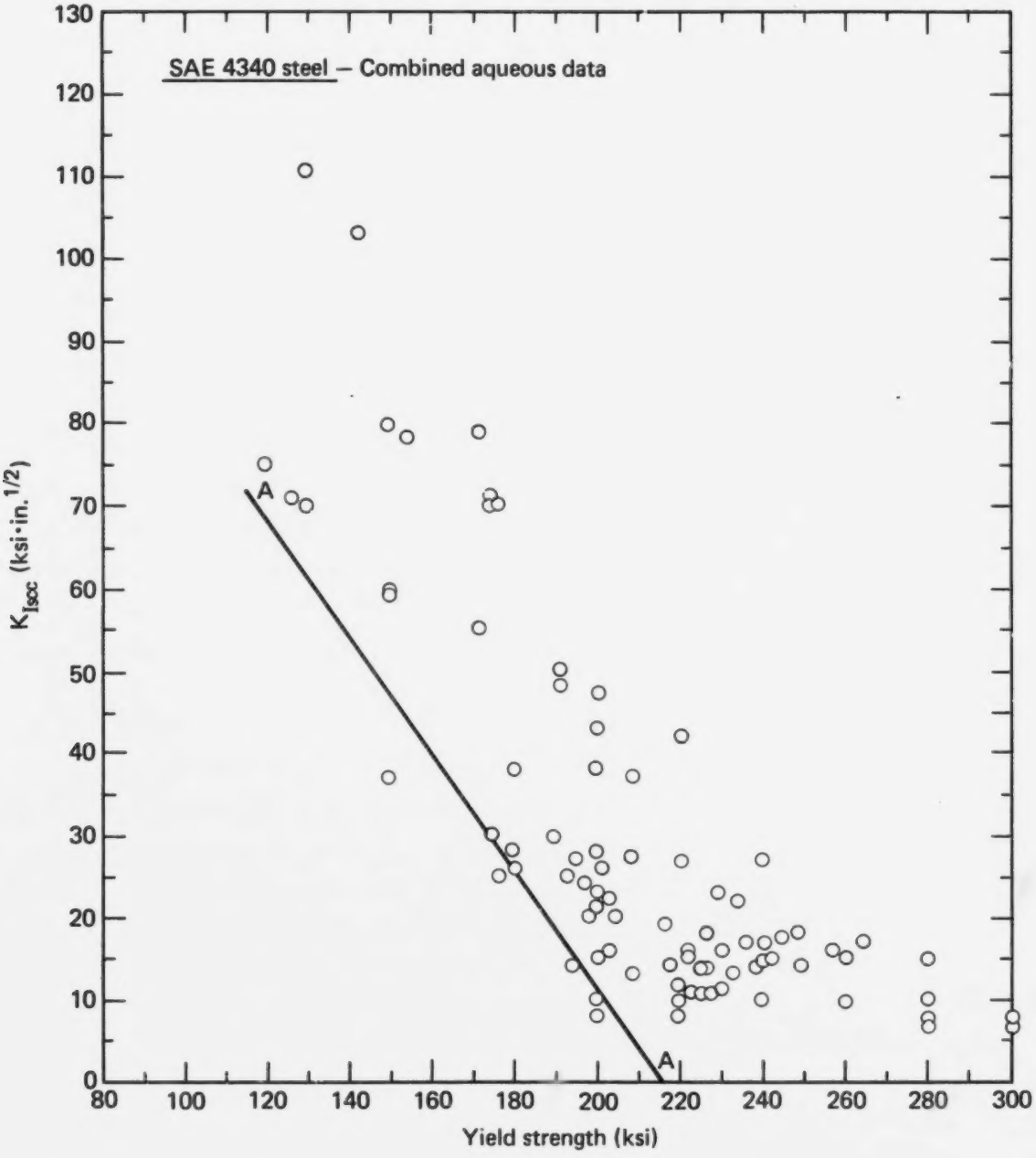

Figure 1. K_{Iscc} versus yield strength for SAE 4340 steel reported for "combined" aqueous data.

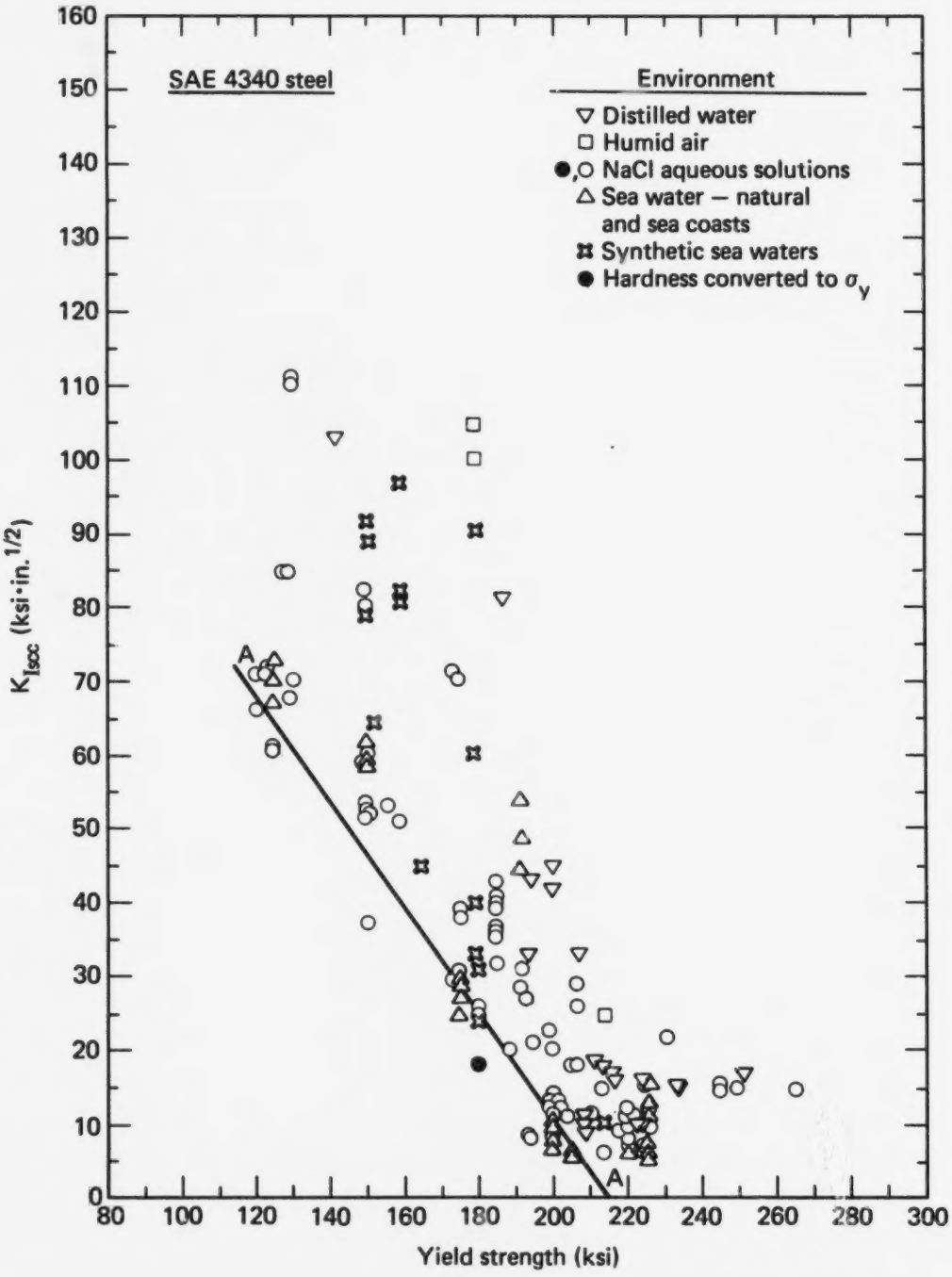

Figure 2. K_{Iscc} versus yield strength for SAE 4340 steel reported for various environments containing water or aqueous chlorides.

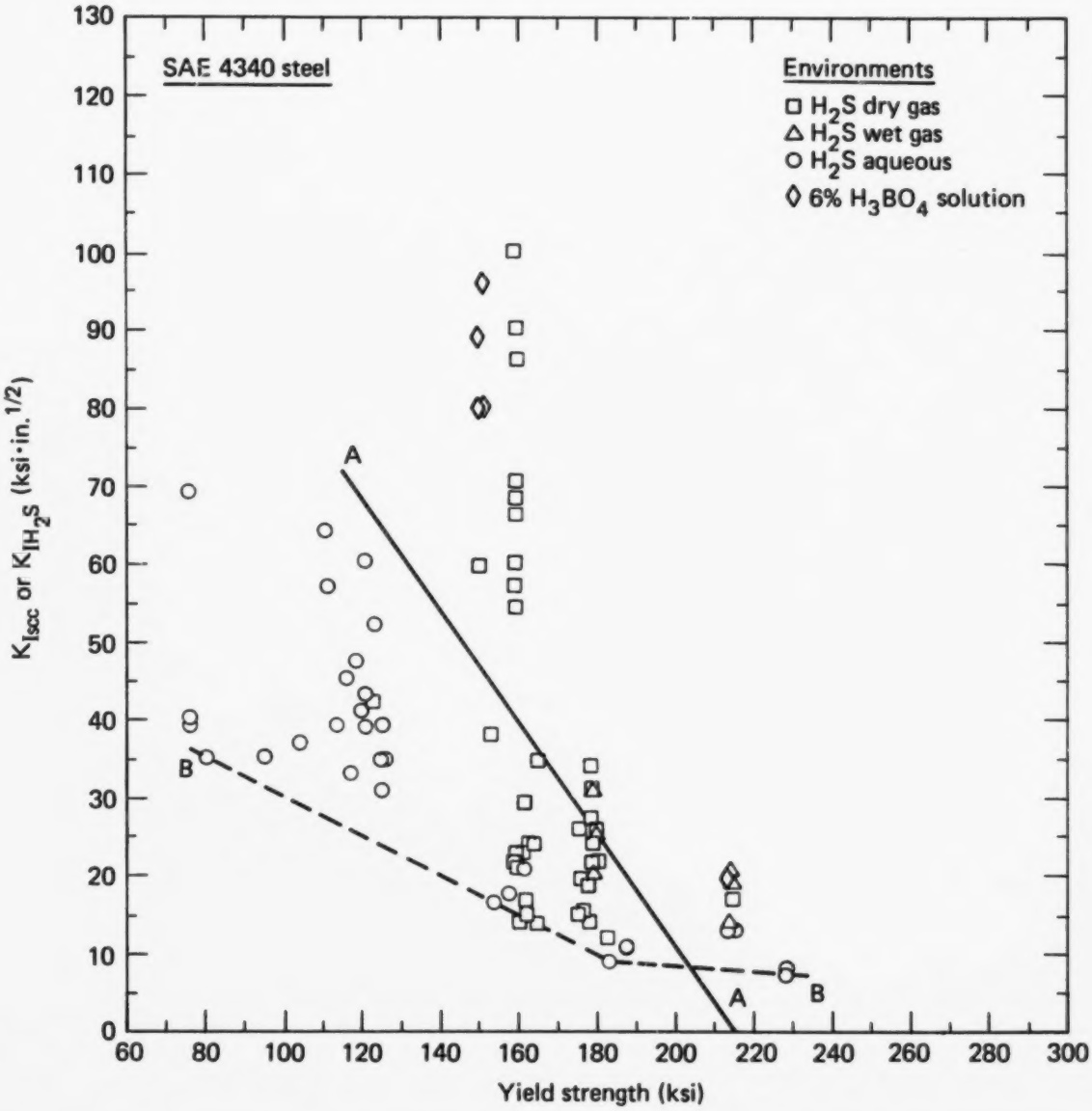

Figure 3. K_{Iscc} and K_{IH_2S} versus yield strength for SAE 4340 steel reported for various H_2S environments and for H_3BO_4 solution.

In both aqueous chlorides and H_2S environments K_{Iscc} appears to be relatively insensitive to σ_y above about 190 ksi, with the lower-bound values falling close to 10 ksi·in.$^{1/2}$. The large scatter reported for the dry gaseous data is primarily related to the range of H_2S partial pressures. For moist H_2S gas, no effect of pressure was obtained over the range from 3 to 115 psi.[65] Also included are data on compact bolt-loaded tension specimens tested in 6% H_3BO_4 aqueous solutions reported by Landerman.[49] Data on 4340 steel exposed to H_2 environments are presented below.

3.2: SAE 43XX-TYPE OF STEELS:

Figure 4 contains data for several different 43XX-type of steels heat treated to ultra-high strengths and exposed to various environments, but excluding sulfides and H_2. The data all fall within the range where K_{Iscc} is insensitive to σ_y. The lower-bound curve, C-C, for these specialty steels at $\sigma_y \geq 190$ ksi lies at about 10 ksi·in.$^{1/2}$. This is higher than the lower-bound values of 5 to 8 plotted for the 4340 steels over the same σ_y range in Figs. 1 and 2. The references for Fig. 4 are 4330M,[4,69] 4330V[4,5,47,48,57] 4335,[53] 4340M and 300M,[4,28,41,47,54,57,59,71] and experimental 4340-(1)[72] and 4340-(2)[4,5,41]. Figure 5 contains limited data for 4335V,[4,73] 4340,[4,12,21,65,74-77] and 300M[78] in H_2 gas. The large range in K_{IH_2} reported for a given σ_y is primarily related to a corresponding variation in the partial pressure of H_2.

3.3: SAE 41XX-TYPE OF STEELS

Figure 6 shows data reported for 4130[5,24,53] and 4140[47,79] tested in H_2O and aqueous chlorides. The data are very limited; nevertheless, the results confirm the conclusion drawn from the corresponding 4340 data that the proposed NRC lower-bound curve is reasonable and that the K_{Iscc} value is insensitive to variations in σ_y above about 190 ksi. Data on the effect of H_2S environments for 4130,[48,81-83] 4130 modified,[83] 4135 modified,[68,80,82-84] and 4140[4,5,61,66,67] are shown in Fig. 7. Figure 8 contains some data on hydrogen gas effects.[4,85,86] As in the previous section, the large spread in K_{IH_2} is due to a corresponding spread in hydrogen pressure,

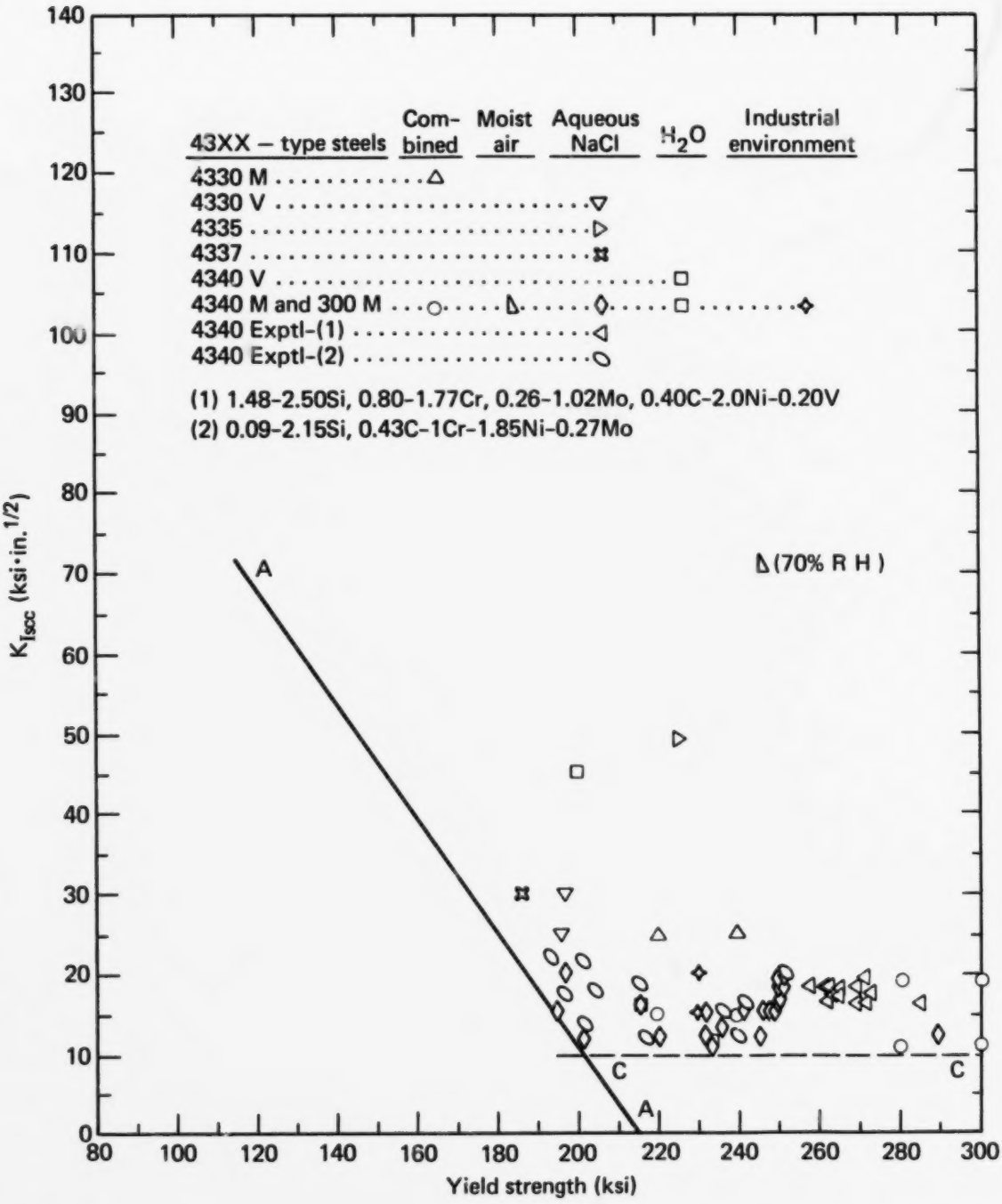

Figure 4. K_{Iscc} versus yield strength for several high-strength heat-treated SAE 43XX-type steels reported for various types of environments.

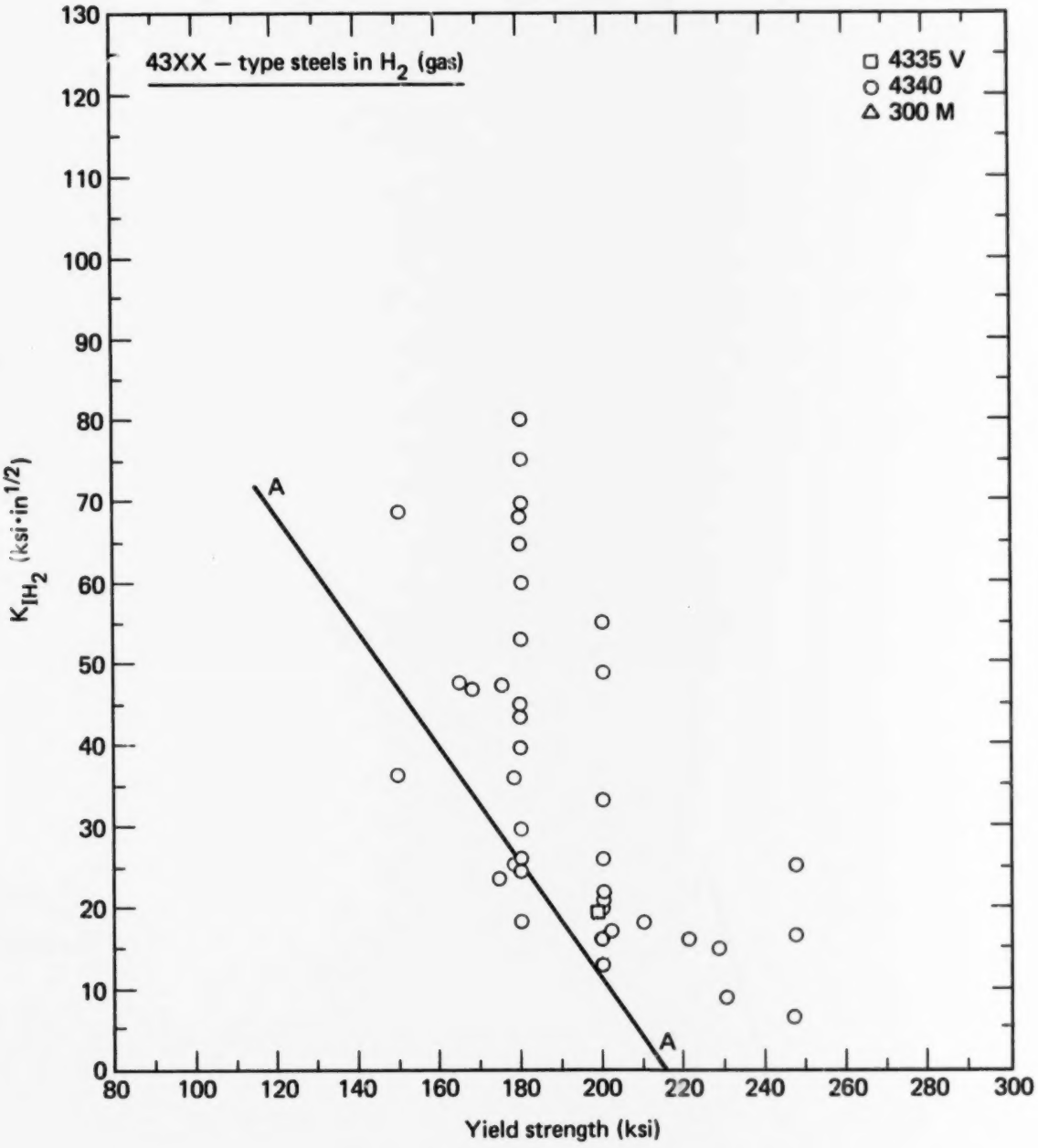

Figure 5. K_{IH_2} versus yield strength for SAE 4335V, SAE 4340, and 300M steels reported for H_2 gas.

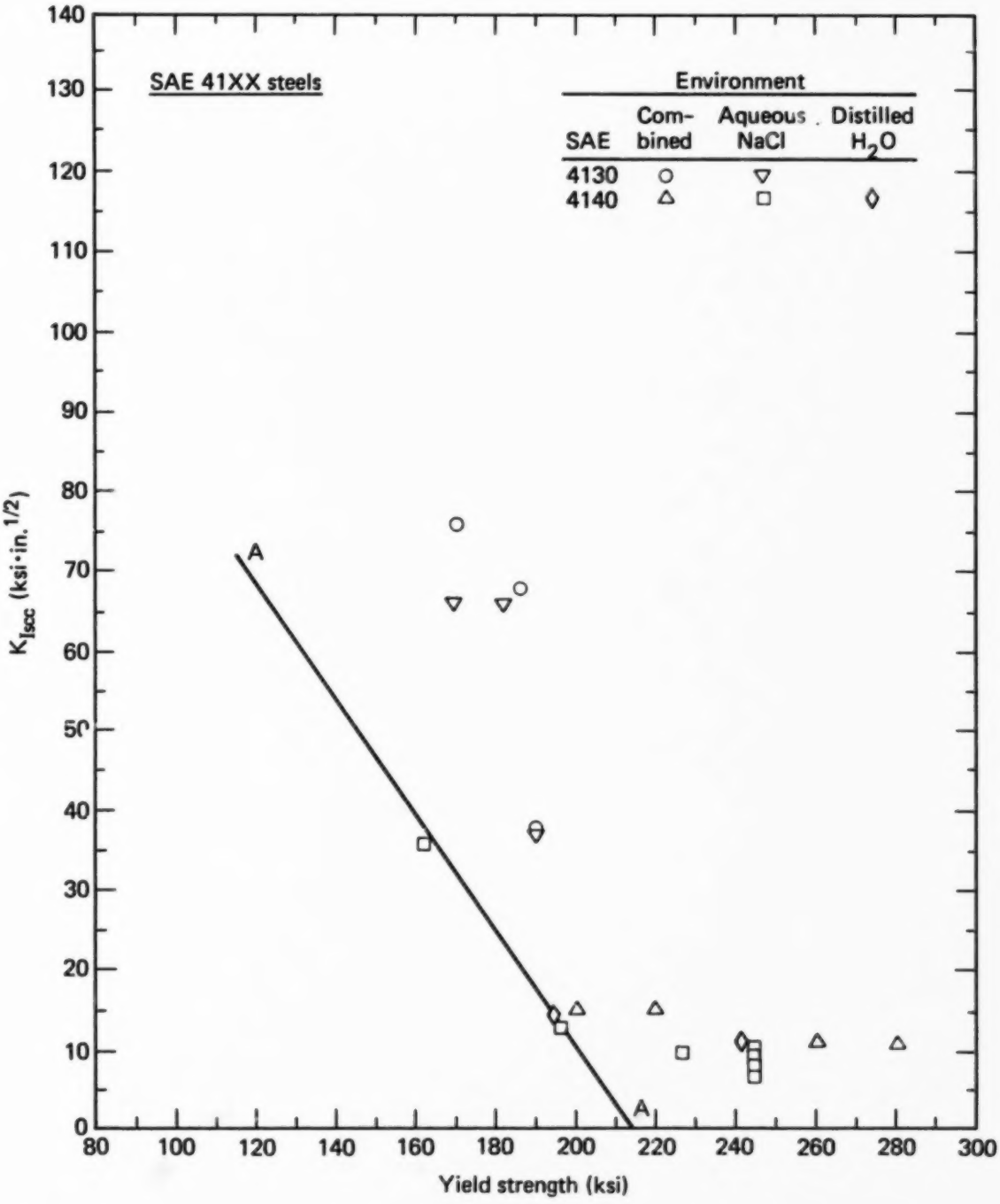

Figure 6. K_{Iscc} versus yield strength for SAE 4130 and SAE 4140 steels reported for "combined," aqueous NaCl, and distilled H_2O environments.

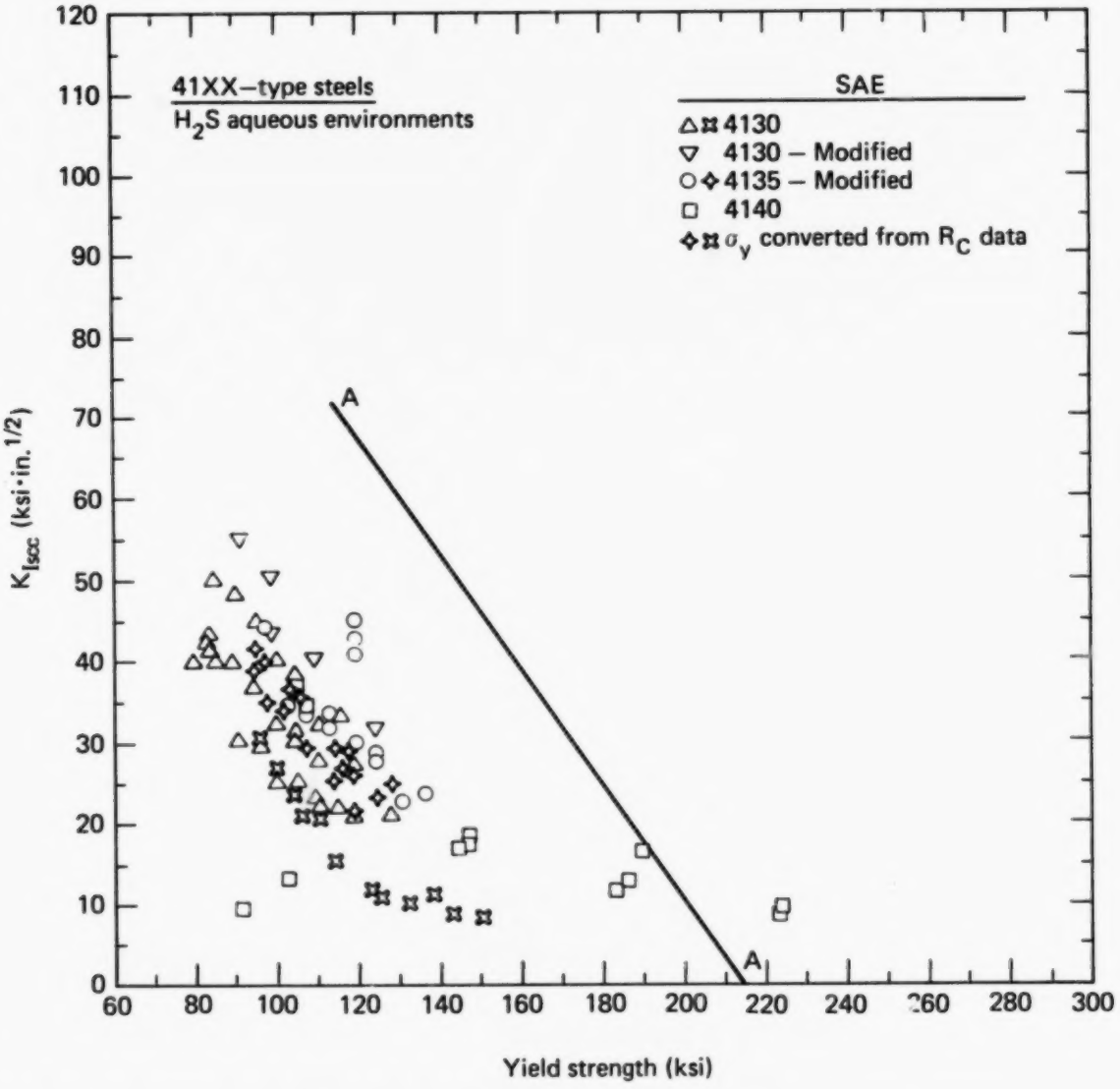

Figure 7. $K_{I\,scc}$ versus yield strength for several SAE 41XX-type steels reported for H_2S aqueous environments.

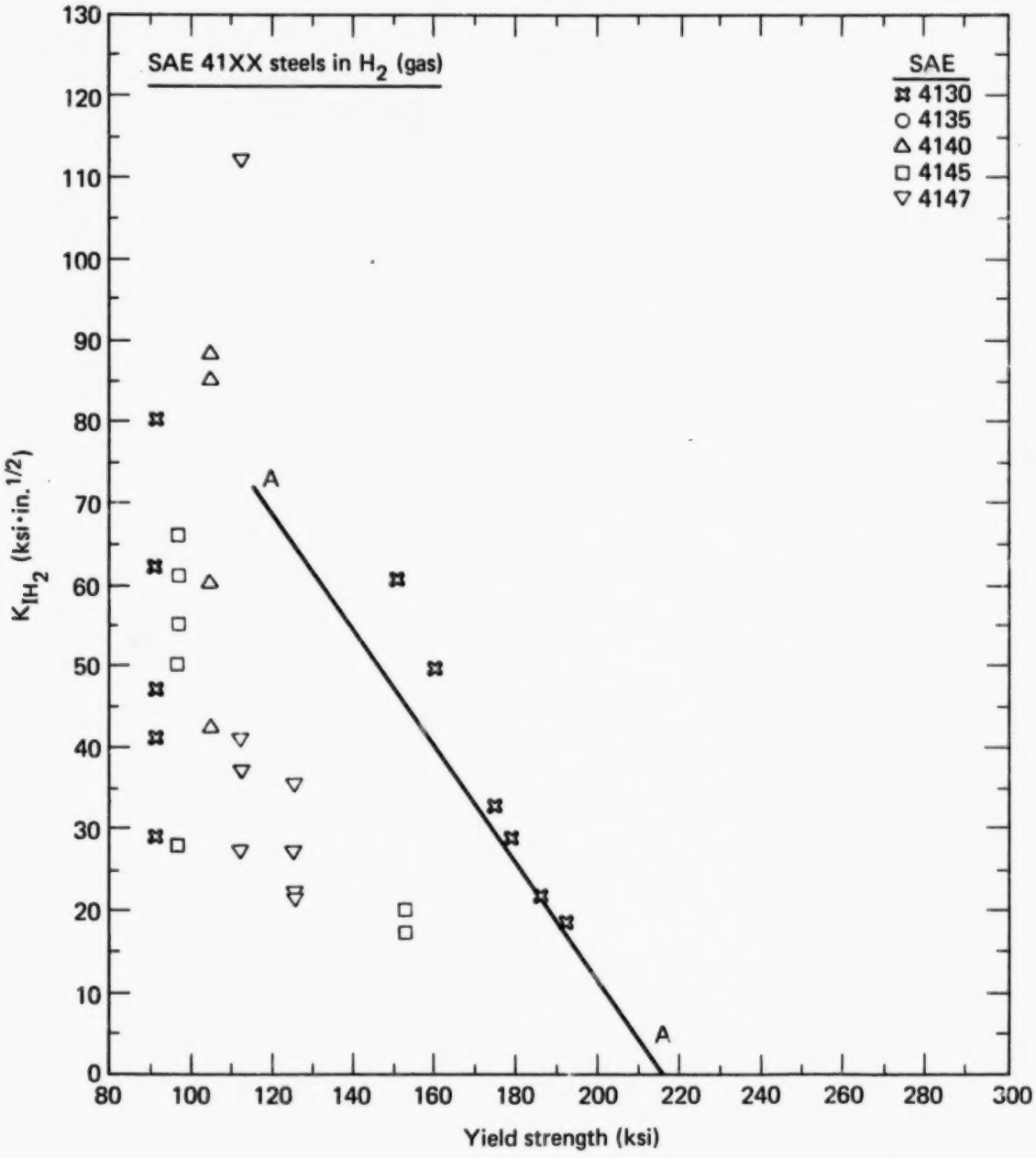

Figure 8. K_{IH_2} versus yield strength for several SAE 41XX steels reported for H_2 gas.

which ranges from about 11 to 14,000 psi. The data for 4130 that lie just above the proposed NRC lower-bound curve, A-A, were obtained at 11.2 psi.[86] Those data falling significantly below this curve generally were obtained from tests at above 3000 psi H_2 pressure.[85]

3.4: D6AC STEEL

Figure 9 contains available data for D6AC exposed to "combined," distilled water, and aqueous chloride environments,[4,5,17-20,28,29,34,37-39,42,45,47,53,56,59,87-90]. All data clearly lie above the proposed NRC lower-bound curve, A-A. However, at the higher stress levels, above about 220 ksi, the spread in K_{Iscc} as well as the lower-bound values appear to be independent of σ_y. The data for D6AC are not too dissimilar from those obtained for the 43XX and 41XX series of steels.

3.5: H-11 DIE STEEL

Figure 10 shows data plotted for H-11 die steel tested in humid air and the various aqueous environments of water, sea water, coastal, and NaCl.[4,5,29,35,37,38,56,57,59,91,92] The comments made for the D6AC steel data are also applicable here. The data indicated by the diamond symbols, from low to high K_{Iscc} values, correspond to variations in humidity from 100 to 0.1%, respectively.

3.6: HY STEELS

Figure 11 contains the results reported for HY steels (HY80 to HY150) in air, aqueous chloride, synthetic and natural sea-water environments,[93-104] and in combined environments.[104-110] The arrows on four of the data points indicate "invalid" data due to insufficient section size. With reference to the proposed NRC lower-bound curve, A-A, these steels are superior to the 43XX and 41XX steels at least for $\sigma_y \leq 170$. Bolts, however, are usually not made of HY-type steels. Some data are included for H_2S environments[4,67] which fall below the proposed NRC lower-bound curve for non-H_2S environments.

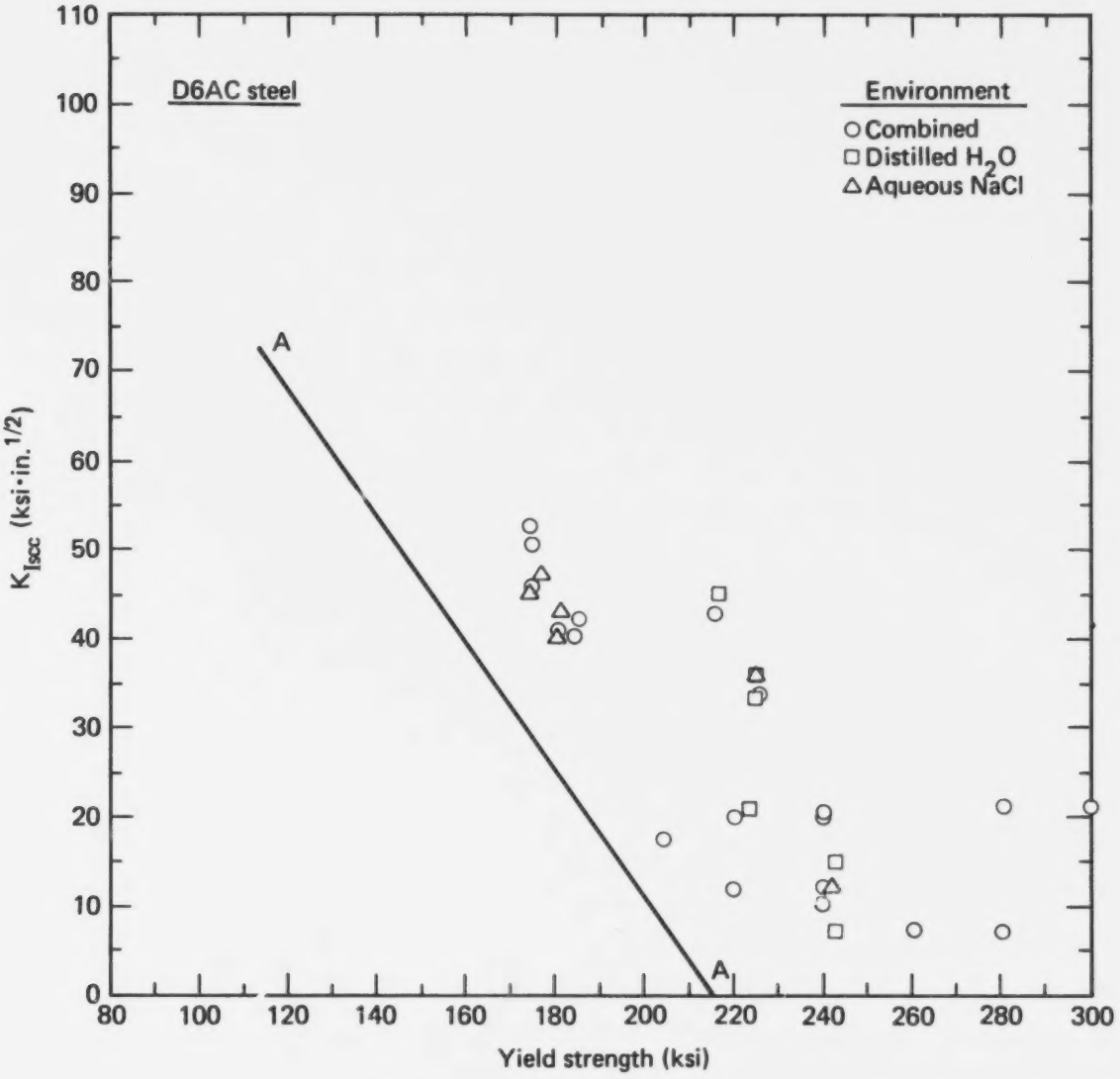

Figure 9. K_{Iscc} versus yield strength for D6AC steel reported for "combined," aqueous NaCl, and distilled H_2O environments.

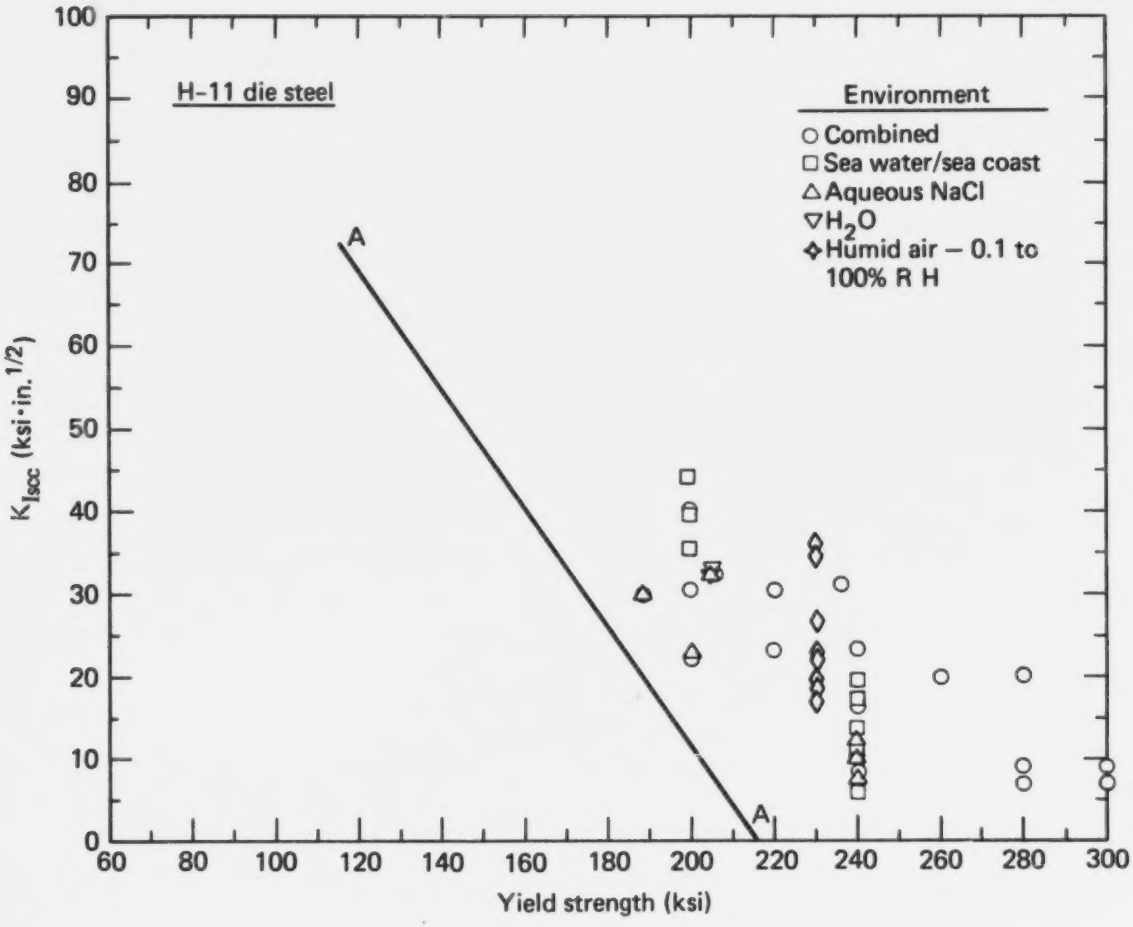

Figure 10. K_{Iscc} versus yield strength for H-11 die steel reported for various environments containing water or aqueous chlorides.

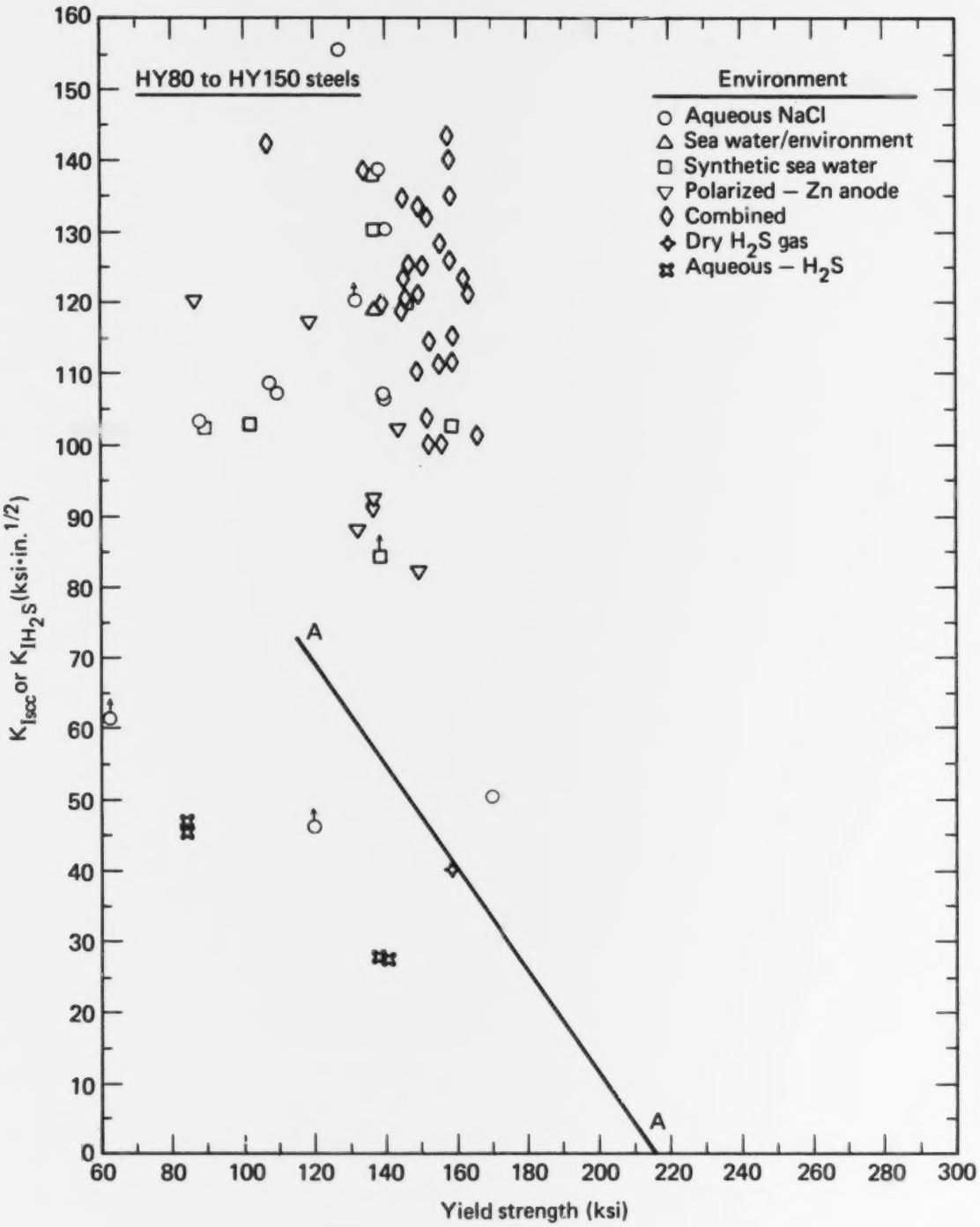

Figure 11. K_{Iscc} versus yield strength for HY steels reported for various environments containing water, aqueous chlorides, and/or H_2S.

3.7: MISCELLANEOUS LOW-ALLOY STEELS

Limited data were obtained for various miscellaneous low-alloy steels tested in water and various chloride-containing environments. These data are primarily for foreign steels. The data are plotted in Fig. 12.[4,15,28,95,102,111-117] With the exception of two questionable data points indicated by arrows the data either fall close to or above the proposed NRC lower-bound curve. Again, the lower-bound data appear to be independent of σ_y above a value of about 190 ksi.

Figure 13 contains data available for miscellaneous low-alloy steels tested in dry H_2S gas[4,63,115,118,119] and in aqueous H_2S solutions,[4,67,68,82,101,119-124] (usually the H_2S saturated, 3.5% NaCl-0.5% acetic acid NACE solution). The lower-bound curve for H_2S environments would fall considerably below the proposed NRC curve, A-A.

Threshold data of several low-alloy steels exposed to H_2 gas[4,85,115,125-128] are plotted in Fig. 14. Also included are some results for HY80, HY100, and HY130[4,85,125] and H-11 die steel.[129] As in previous such plots the range in K_{IH_2} for a given stress level is usually related to the P_{H_2}. The P_{H_2} for the plotted data ranges from 14.7 to 14,000 psi. It would be more informative if the data for all the low-alloy steels were analyzed as a function of P_{H_2} and then cross-plotted with K_{IH_2} versus σ_y for a constant P_{H_2}. However, since the H_2 and H_2S results were peripheral to the main objective of this review, no further effort was undertaken in this area.

3.8: PRECIPITATION HARDENING STAINLESS STEELS

Bolting materials of Custom 455 and 17-4 PH are being used in the reactor plants supports.[1,2] Data obtained for these two materials are plotted in Figs. 15[4,49,60,130,131] and 16,[4,5,17,19,47,52,53,130,132-137] respectively. The proposed NRC lower-bound curves for both the low-alloy steels (A-A) and maraging steels (D-D) are superimposed on these data. The lower-bound curve, E-E, in Fig. 15 corresponds to the limited data for H_2S. Excluding the H_2S data, the lower-bound curve for the remaining environments

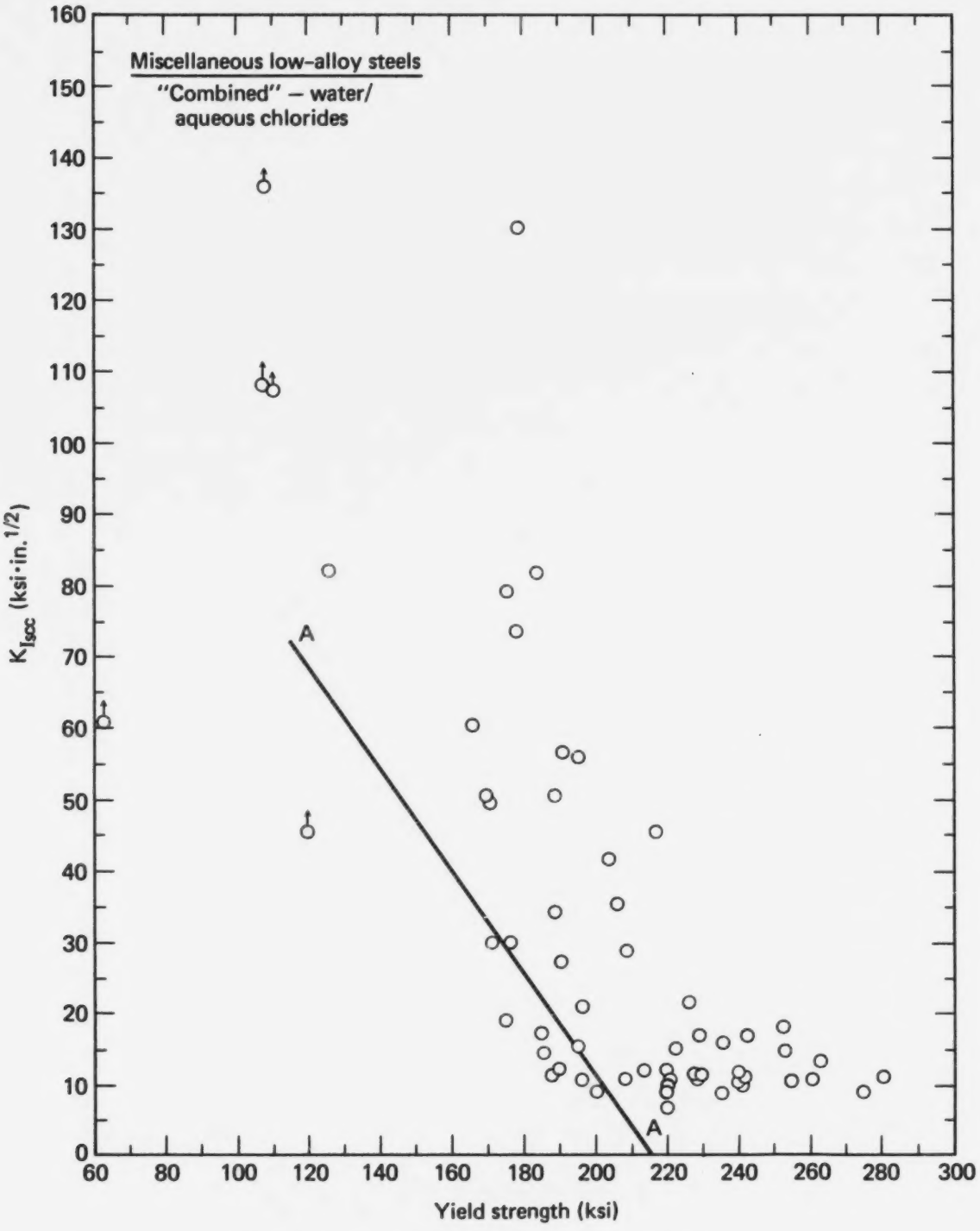

Figure 12. K_{Iscc} versus yield strength for miscellaneous low-alloy steels reported for "combined" environments.

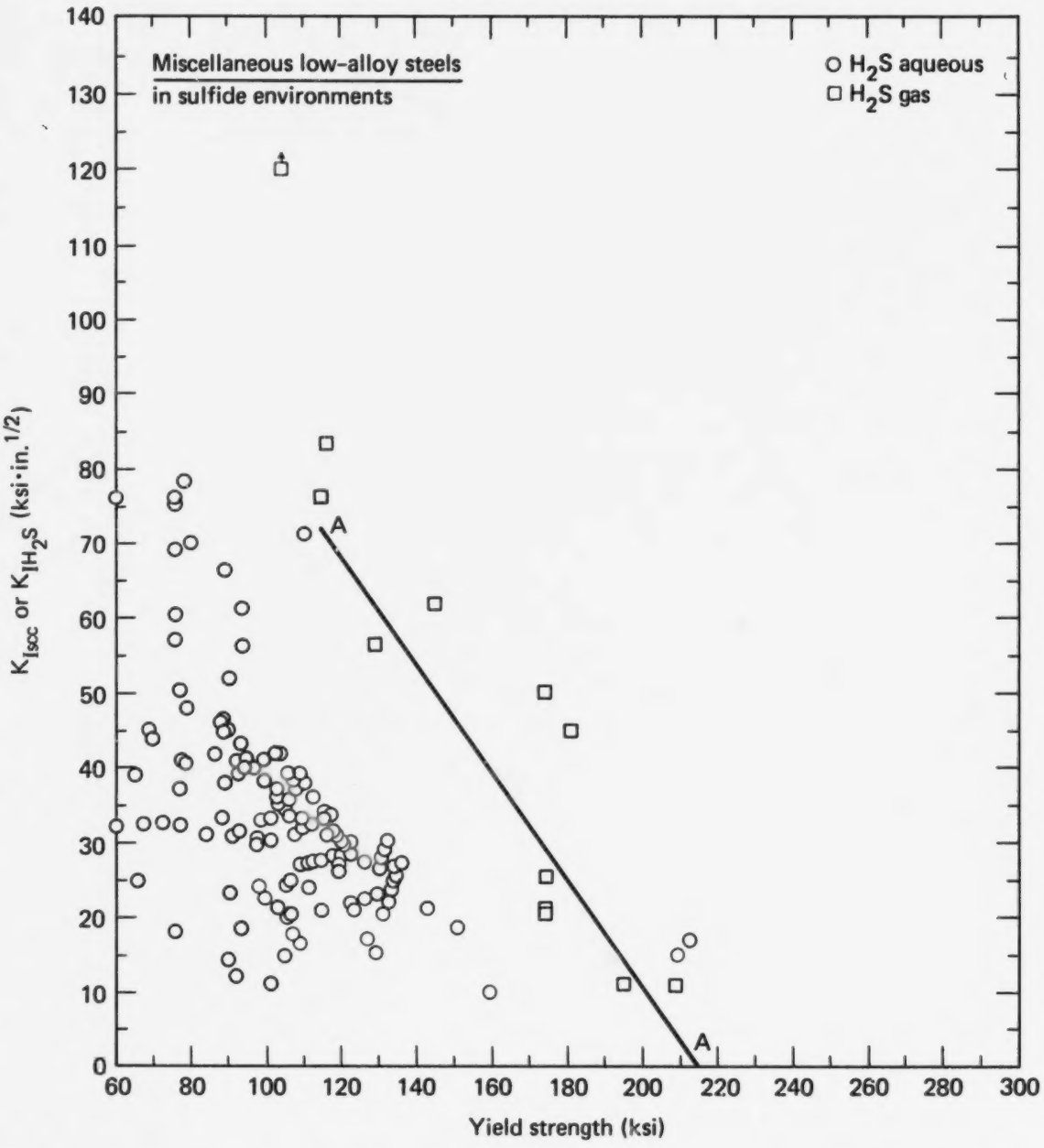

Figure 13. K_{Iscc} versus yield strength for miscellaneous low-alloy steels reported for H_2S environments.

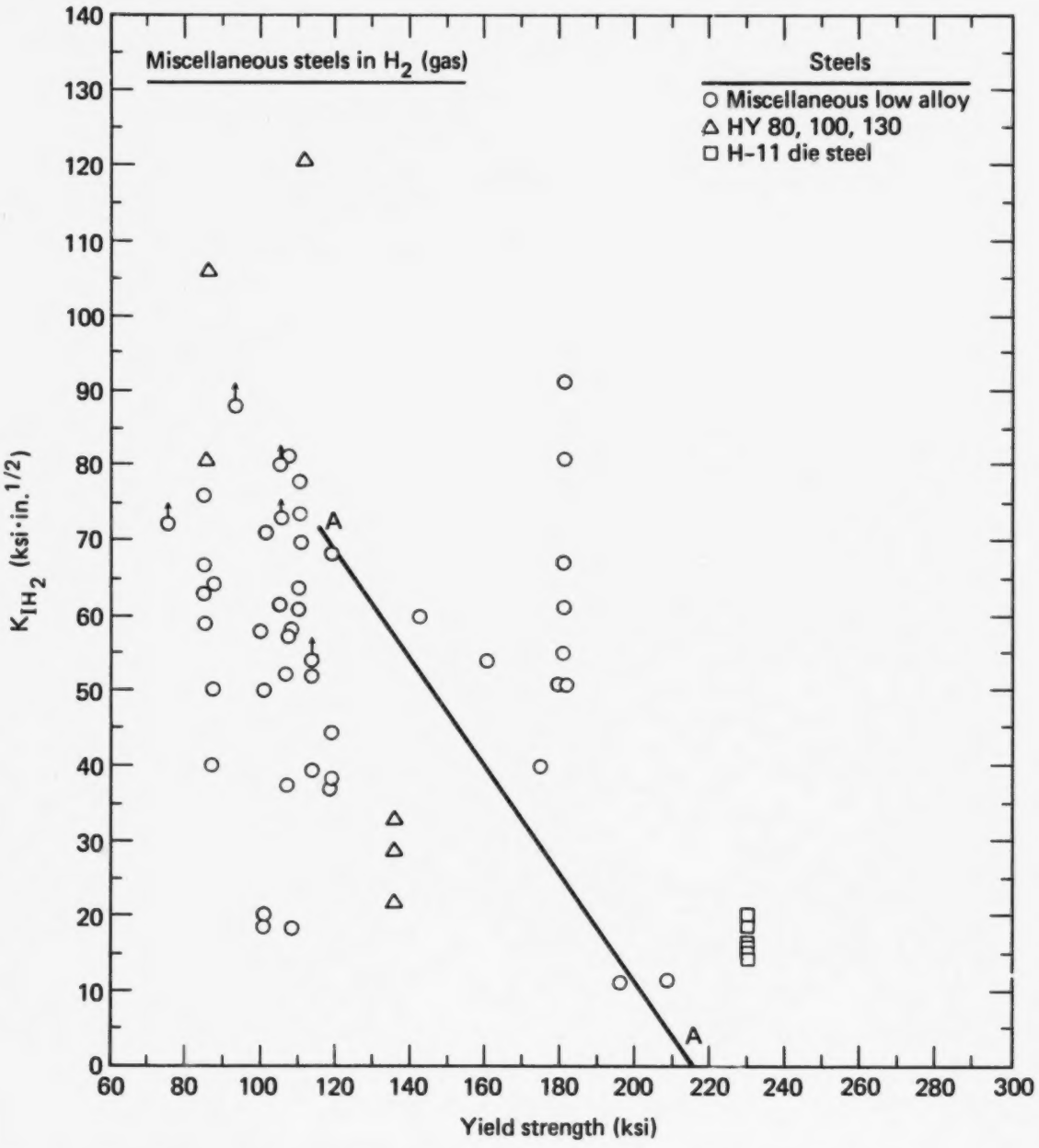

Figure 14. K_{IH2} versus yield strength for several different types of steels reported for H_2 gas.

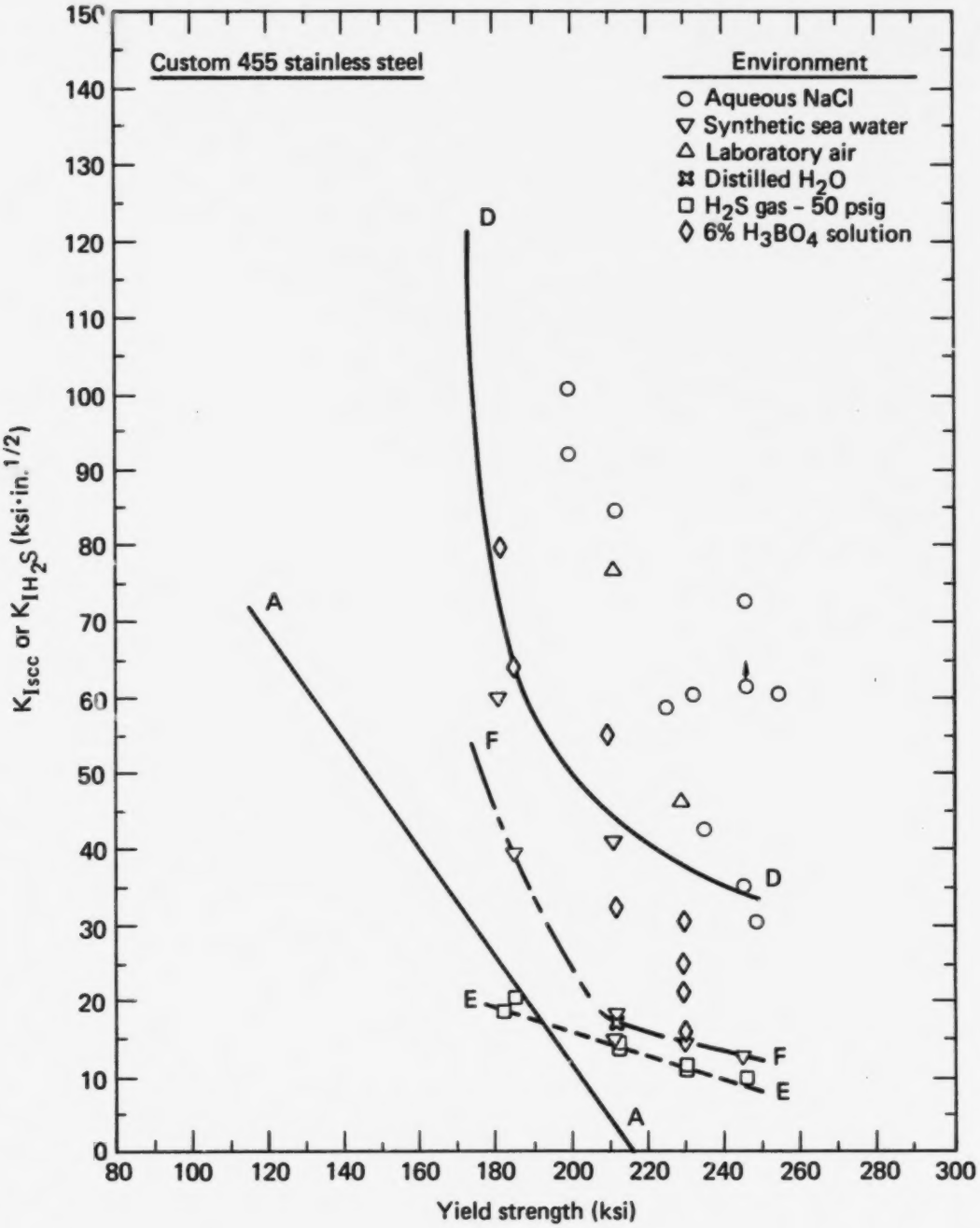

Figure 15. K_{Iscc} or K_{IH_2S} versus yield strength for Custom 455 stainless steel reported for various environments containing water, aqueous chlorides, air, H_2S gas, or H_3BO_4 solution.

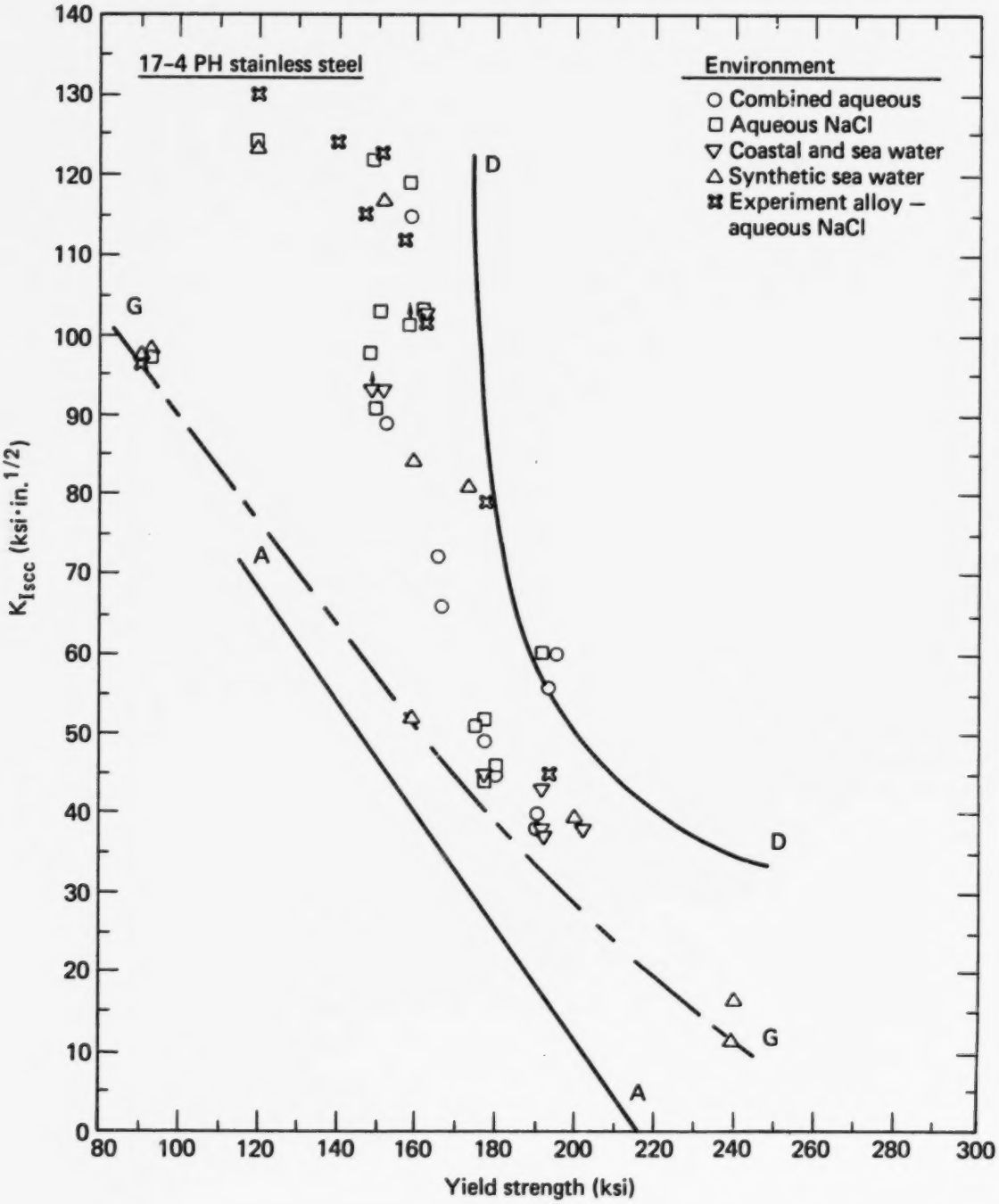

Figure 16. K_{Iscc} versus yield strength for 17-4 PH stainless steel reported for "combined" or specific aqueous chloride environments.

falls between the curves for the low-alloy and maraging steels as suggested by curve F-F. With the exception of the aqueous NaCl data, most of the data used for Fig. 15 were obtained from reference 49. Consistent with the data for Custom 455, the data obtained for 17-4 PH suggest that the lower-bound curve for this alloy also falls between the curves for the low-alloy and maraging steels. This is also depicted by curve G-G in Fig. 16. The stress level at which K_{Iscc} becomes insensitive to σ_y for these two steels is not clearly evident. Excluding the H_2S gas, it would appear to be relatively insensitive to σ_y above 210 ksi.

3.9: 18Ni-MARAGING STEELS

Considerable information is available on SCC of 18Ni-maraging steels in various aqueous environments. Figure 17 contains K_{Iscc} data obtained largely from review papers[4,5,17] for the "combined" (water and chloride-bearing) environments.[4,5,17,19,20,26,37,40,42,43,45, 46,59,133, 138-154] Clearly, the proposed NRC lower-bound curve, D-D, for maraging steels should be displaced to considerably lower levels at $\sigma_y \geq 190$ ksi, namely to H-H. Figure 18 contains data plotted for specific environments.[4,18,21,27-29,40,47,57,58,60,71,102,114,138,142,143,150,151,155-160] Included also in Fig. 18 are the proposed NRC lower-bound curve, D-D, the curve H-H based on the data in Fig. 17, and data on a number of experimental 18Ni-maraging steels.[29,151,153,157,161]

Because of the sharp drop in K_{Iscc} with increasing σ_y as well as the large scatter in these values, considerable care must be taken in the use of maraging steels for nuclear-reactor bolting materials. It would appear that samples from the same stock as the heat-treated material actually in use should be subjected to qualification SCC tests to evaluate its reliability in the reactor environment. In fact, the large scatter in virtually all the plotted data suggests that this procedure should be recommended for all steels.

4. DISCUSSION ON INFLUENCING FACTORS

The plotted data clearly show that a considerable spread in K_{Iscc} is obtained at any given σ_y for a given material or type of material. We will

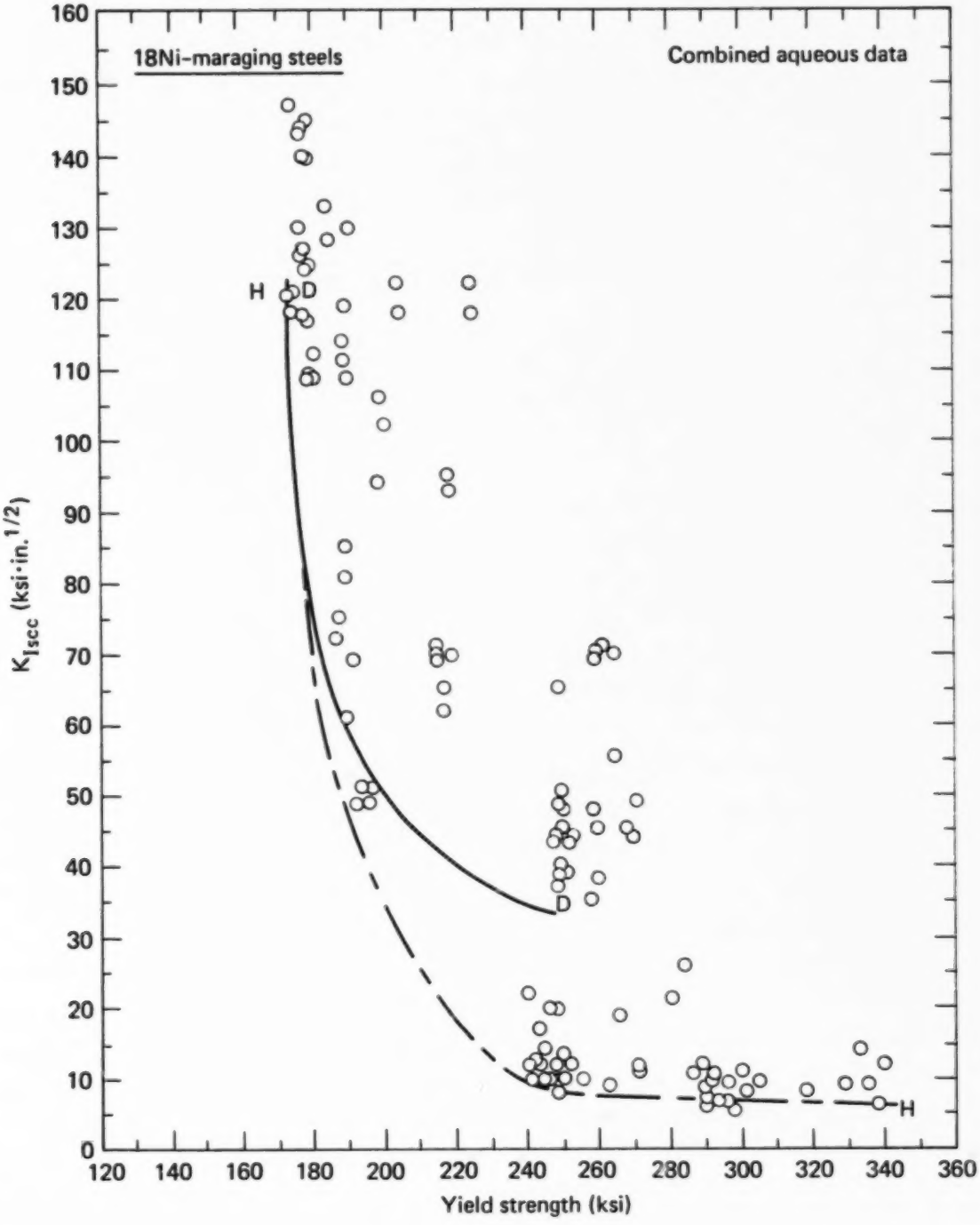

Figure 17. K_{Iscc} versus yield strength for 18Ni-maraging steels reported for "combined" aqueous environments.

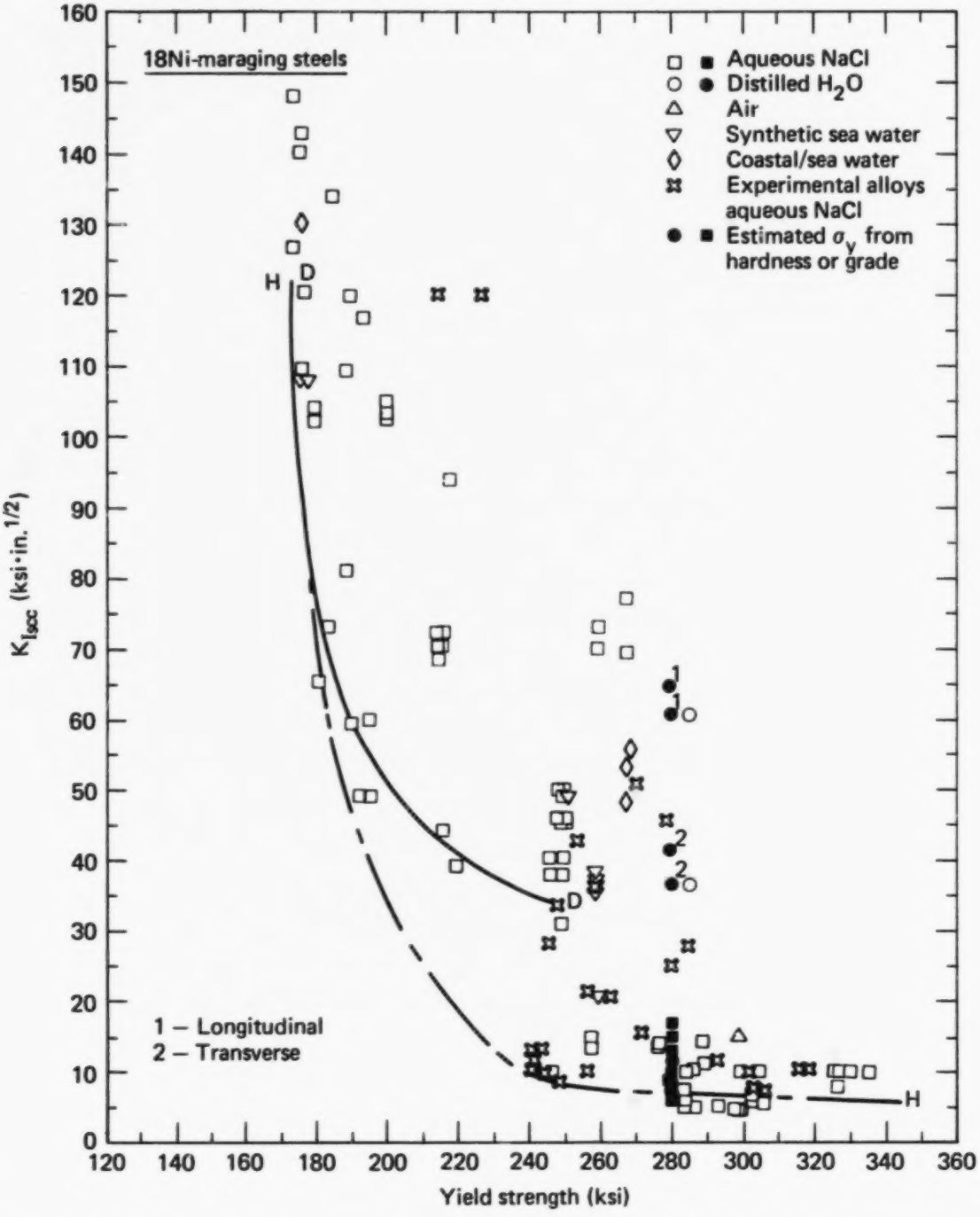

Figure 18. K_{Iscc} versus yield strength for 18Ni-maraging steels reported for water, air, and various aqueous chloride environments.

attempt here to examine the factors that may result in such large variations in the data. The effects of material variables associated with melting practices,[4,5,157,162] heat treatment and thermomechanical processing,[4-6, 28,29,31,48,53,80,84,88,112,157,162-164] and composition[4-6,12,21,25,29, 41,48,54,56,64,66,73,80,82,83,151,157,164], and environmental parameters involving pH,[4-6,48 88,157] polarization potential,[4-6,11,12,43,48,53, 150,165-168] cathodic poisons,[4-6,53] and temperature[4-6,48,86,92,169,170] on SCC are discussed in a number of articles. (No attempt was made here to make a complete listing of such references). The effects related to material variables often are unclear and controversial; by contrast, the effects of environmental parameters are mostly well established. Several examples illustrating these comments will be given here. We will not discuss the effects of variations in specimen configurations and testing procedures. The reader is referred to several detailed papers on this subject.[47,51,93, 103,171-174] In the discussions that follow, we will refer primarily to H_2O and aqueous chloride environments.

4.1: COMPOSITIONAL EFFECTS

Sandoz, in one of his review articles,[5] points out that the wide range in K_{Iscc} is a reflection of the many variables in melting practice, processing, fabrication, and heat treatment. The effect of uncertain variables in testing further widen this range. He further states[12] that there are no fundamental effects of composition on K_{Iscc}. However, above K_{Iscc} the CGR can be significantly influenced by composition. For a given K_{Iscc} and σ_y the CGR is lower[12] and time to failure longer[29] in the more highly alloyed high-strength steels. Improved melting practices, with better control of impurities, have resulted in significant increases in the K_{Ic} values, especially for the intermediate strength steels.[5] However, the corresponding effect on K_{Iscc}, although somewhat similar, is considerably less; in fact, factors such as composition and heat treatment that benefit K_{Ic} may adversely affect K_{Iscc}. Carter points out that for aqueous H_2S there is no apparent correlation between K_{Iscc} and K_{Ic}.[4] Waid and Ault studied a series of low- and medium-alloy steels and found that, although compositional variations affected K_{Ic}, the K_{Iscc} in aqueous NaCl was insensitive to these variations.[72] However, such compositional changes

can have marked influences on CGR. A number of investigators have pointed out that variations in Si content do not affect K_{Iscc}; however, an increase in Si may markedly decrease the CGR.[4,41,54,72] This affect has been attributed to the retardation by Si in the replacement of ε-carbide with the high-temperature carbide during tempering.[41] The presence of retained austenite and ε-carbide in 4340 and 300 M steels was observed to reduce the CGR in aqueous NaCl without affecting K_{Iscc}.[54] For 35NCD16 (4Ni-2Cr-0.5Mo-0.35C), also tested in aqueous NaCl, retained austenite did not show any effect on K_{Iscc}, but it did reduce the CGR.[112] Carbide-forming elements have little affect, if any, on K_{Iscc}; but, their presence apparently increases the incubation time and time to fracture as evidenced by tests in distilled H_2O.[4] Reduction of P and S improves the resistance to crack growth, but have little effect on K_{Iscc}.[41,48] In maraging steels compositional effects are at most marginal.[4,151,157]

A reversal in the general observation of an increase in K_{Iscc} with alloying was attributed to the greater degree of twinned martensite in a 9Ni-4Co-0.45C steel as compared to that present in the low-alloy steels.[72] The twinned martensite is more succeptible to hydrogen attack than is the untwinned martensite.[48] Carter points out in his extensive review paper[4] that the effect of alloying elements are contradictory and the only consistant trend is that shown by carbon; i.e., an increase in C causes a decrease in the resistance to SCC. But, as pointed out below, an anomalous behavior is obtained for the highest carbon content. The trend with C content is consistent with the observation that low-carbon martensites are less susceptible to SCC and hydrogen attack than are high-carbon martensites.[5] In a plot of K_{Iscc} versus σ_y for aqueous NaCl, Carter[4] shows the data for 4130, 4330V, and 0.2C steels superimpose on each other; but, these data fall significantly above those for a 4340 steel plotted in the same figure.

In Fig. 19, the data published by Sandoz[25] show the effects of variations of several elements on the SCC of essentially 43XX-type steels in 3.5% NaCl. Carbon and Mn are the only two elements showing definite trends. There is a considerable drop in K_{Iscc} from 0.2 to 0.3C. This is inconsistant with similar results reported above by Carter[4] for of the 0.3 and 0.2 carbon contents, unless one considers the effects of alloying

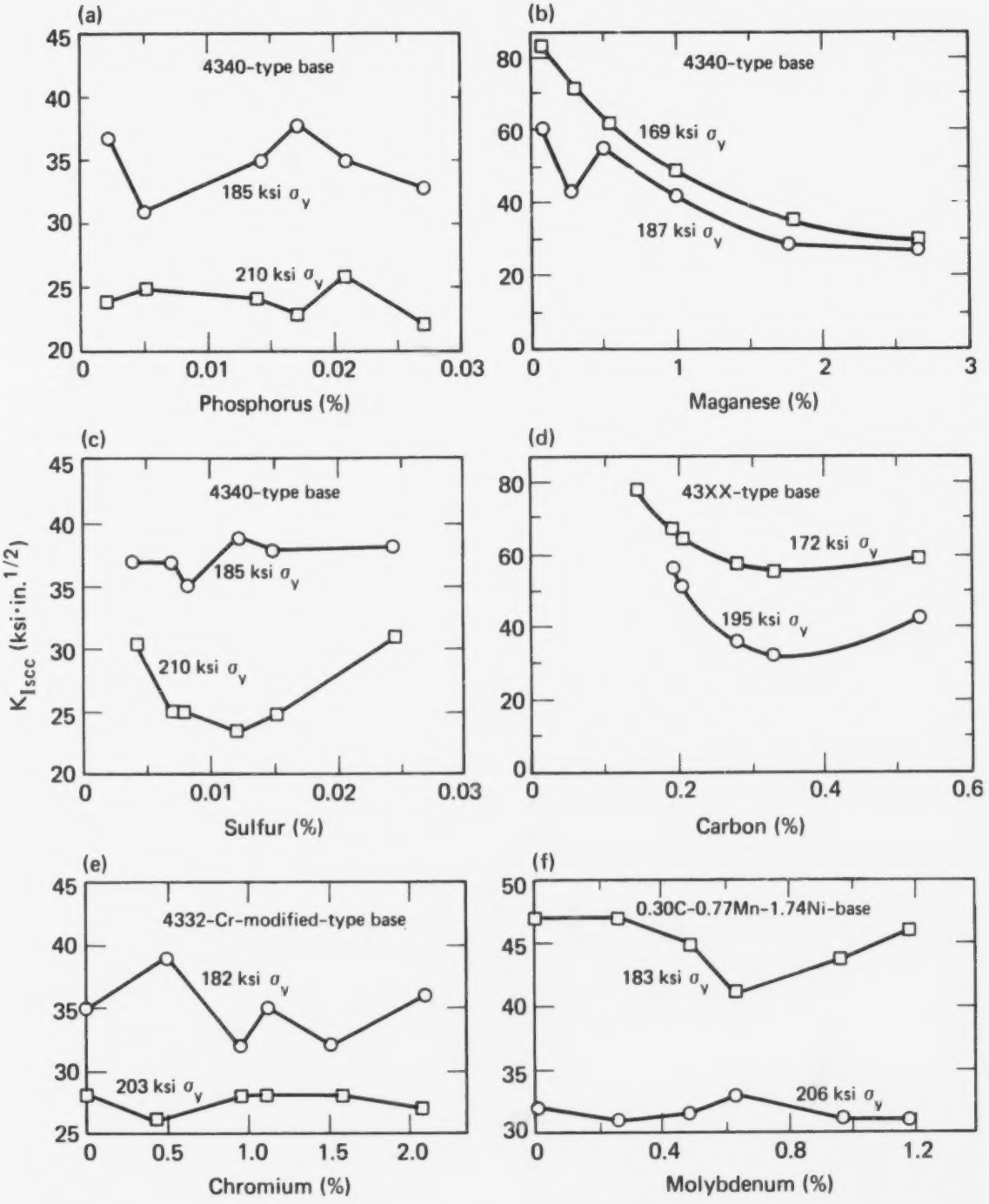

Figure 19. Effect of various impurities and alloying elements on K_{Iscc} for ASE 43XX-type steels.[25]

counteracting the carbon effect. However, the 4130 and 4330V steels also show identical results. It is suprising that K_{Iscc} should exhibit a minimum and then increase with carbon content above 0.35C. Furthermore, this carbon content corresponds approximately to where there is a transition from untwinned to the more hydrogen-susceptible twinned martensite. We believe that the two single values at 0.53C may be suspect or are affected by other compositional factors.

Carter[4] points out that the trend shown here for Mn in the 4340 steel is in conflict with the insensitivity of Mn content on SCC in the lower carbon steels. He further points out that the 4340 data may reflect a secondary effect, in that Mn lowers the $A_{1,3}$ temperature and, in tempering, this temperature may inadvertedly have been exceeded resulting in some reversion to austenite, especially if segregation is present. This would result in regions of untempered martensite causing a reduction in K_{Iscc}.[4] Alternatively, Sandoz[21] attributes the effect of C and Mn on K_{Iscc} to the crack tip changing from relatively anodic to cathodic with an increase in content in either of these two elements. Both explanations may be justified. It is clear that the effects of impurities and alloying elements on K_{Iscc} are not too well understood.

4.2: MICROSTRUCTURAL EFFECTS

Variations in heat treatment, prior deformation, and thermomechanical processing, all show effects on K_{Iscc} through the microstructures that are developed. An analysis of the review paper by Carter[4] suggests that there are fewer contradictions on the effects of heat treatment and microstructure, as well as of composition, for exposures in sulfide and hydrogen environments then there are for H_2O and aqueous chloride environments. For the low-alloy steels there appears to be little or no effect of austenite grain size on K_{Iscc}[4,5,6,29,31,48,53,88] but an increase in grain size increases CGR and decreases t_f. In maraging steels the role of grain size is not at all clear; annealing may cause other factors to enter, such as grain boundary precipitates and residual elements in solid solution. Furthermore, subsequent aging temperatures may cause opposite or dissimilar effects on K_{Ic}, K_{Iscc}, and CGR. Compared to a normal 500°C aging treatment, underaging may

increase K_{Ic} and decrease K_{Iscc} with an expected corresponding increase in CGR. By contrast, overaging may increase both K_{Ic} and the resistance to SCC.[4,157] Other work has shown that aging temperature has little effect on the K_{Iscc} of maraging steels but can cause significant changes in CGR.[5,29] Excessively high annealing temperatures and poor melting practices will result in a loss in resistance to SCC, believed to be due largely to the precipitation of a Ti(C,N) grain boundary film.[157] Stavros and Paxton,[150] in comparing the properties of an 18Ni-300-grade maraging steel heat-treated to produce 2 different grain sizes (ASTM Nos. 0 and 9), found that there was no significant effect of either grain size or subsequent aging tratment on K_{Iscc}. Values all fell within the range of 10 to 15 ksi·in.$^{1/2}$. However, both the larger grain size, containing grain boundary precipitates, and the lower aging temperatures result in the shortest times to failure. All specimens were heat-treated and aged to the same hardness. It is likely that no grain size effect was seen because the data fall in the σ_y-insensitive region of the K_{Iscc} versus σ_y plots. More meaningful information on K_{Iscc} versus grain-size effects should be obtained for the lower strength maraging grades.

Large differences in K_{Iscc} between longitudinal and transverse orientations have been observed in banded structures.[28] In homogeneous structures there is usually little orientation effect.[4] In general, the proper combinations of deformation and thermal treatments should increase K_{Iscc}. Kerns, et al.[6] review work that shows improvements obtained by ausforming over conventional heat treatment in both K_{Ic} and in the resistance to SCC in distilled H_2O. Most bolting steels are used in the quenched-and-tempered condition. There is some controversy over the relative merits of tempered bainite versus tempered martensite and this may be due largely to environment. In the low-alloy steels the tempered-martensitic structures, relative to normalized-and-tempered or tempered-bainitic structures, appear to have the best resistance to SCC in aqueous sulfides.[80,162] In water and aqueous chlorides no significant effect is obtained for steels with $\sigma_y \geq 200$ ksi[48]. In a 3.5% NaCl environment a 4340 martensitic structure showed a 15-fold increase in CGR relative to that obtained for a lower-bainitic structure, both tempered to approximately 200 ksi σ_y level.[6] The difference in resistance to SCC for the two structures

was attributed to more affective hydrogen trapping in the coherently twinned interfaces in the martensite as contrasted to the incoherent bainitic laths. The same argument should hold for the H_2S environment for here also the dominant mechanism of cracking is generally believed to be due to hydrogen attack.[4,6,48]

4.3: PRESTRESSING AND PRIOR DEFORMATION EFFECTS

Prestressing has been shown to increase the resistance to SCC[4,27,62,89,159,175] and this has been attributed to residual compressive stresses on unloading as well as blunting of the crack tip.[27,62,175] Prestressing has been shown to increase the incubation and failure times as well as K_{Iscc}. This points out the importance of not using excessive fatigue loads in precracking the test specimen.

By contrast to the effect of prestressing a precracked specimen, prior cold work (prestraining) before specimen preparation may reduce the resistance to SCC.[4,5,103,176] Novak[103] shows inconsistent patterns for the effect of small prestrains on K_{Iscc} for different materials tested in synthetic sea water. Inconsistant trends on prestraining to 5% were also obtained for the threshold stress in aqueous NaCl when comparing the results for HY80 and HY130 steels.[176] Cold work prior to heat treatments involving aging or ausforming results in an increase in K_{Iscc}.[5] Carter[4] states that prior cold work is clearly detrimental to SCC resistance in H_2S. The effects of prior cold work in saline solutions must be more clearly established.

4.4: ENVIRONMENTAL EFFECTS

Although testing in distilled H_2O and in aqueous NaCl may give the same K_{Iscc} values, the CGR may be significantly greater in the latter environment.[4] In comparing results from different environments used on two high-strength steels, McIntyre[115] found the same K_{Iscc} value of ~11 ksi·in.$^{1/2}$ for exposures to H_2S gas, aqueous NaCl, and H_2 gas. But, the CGR decreased significantly in the same sequence. The importance of considering crack-growth rate as well as K_{Iscc} is strongly pointed out by these results.

Carter states that the CGR clearly increases with an increase in temperature, but the effect on K_{Iscc} is controversial.[4,48] Here it is probably related to the temperature sensitivity of pitting. Johnson and Willner show that K_{Iscc} for H-11 steel tested in water is independent of temperature over the range of about 5 to 60°C.[92] Charbonnier, et al.,[112] on testing 35NCD16 steel in aqueous NaCl, also found no effect on K_{Iscc} while the CGR increased with an increase in temperature (20 to 50°C). Simmons, et al.[170] studied the effect of temperature on cracking of 4340 in H_2 and distilled water. They observed that an increase in temperature increased the CGR (stage II) but that all the data merged in stage I. This suggests the independence of K_{Iscc} to both environment (H_2O and aqueous chlorides) and temperature. Carter[4] reports data for 4130 steel in H_2O over the range of 1 to 89°C that show an increase in CGR of nearly 3 orders of magnitude with an increase in temperature; again the data all merge in stage I. Landes and Wei[169] also show a strong temperature-dependence of CGR for 4340 in distilled H_2O. In contrast to these results, for H_2S solutions an increase in resistance to SCC was obtained with an increase in temperature.[4,48].

Carter and Hyatt[48] report that in the absence of complete saturation with H_2O vapor the CGR will decrease with an increase in temperature. Tests on the effect of relative humidity on H-11 steel show that there is a strong dependence of K_{Iscc} on RH. The K_{Iscc} decreases with an increase in RH up to almost 50% RH, and changes little thereafter up to 10% RH. The authors[92] propose that water vapor condenses at the crack tip for values > 50% RH. We suggest that the inverse effect of temperature on CGR is associated also with condensation, in that condensation at the crack tip will occur at higher values of RH as the temperature is raised. The CGR also increases with RH and levels off at somewhat above 50% RH.[92] Simmons, et al.[170] also show a decrease in K_{Iscc} with an increase in RH. Studies on SAE 4340 were made at 0.1, 0.5, and 8 torr water-vapor pressure.

The effect of pH is reflected largely by the solution chemistry at the crack tip, where a pH of about 3 to 4 is reached early in the cracking process, relatively independent of the environmental pH.[13,16] Sandoz[5] in considering "salt waters", states that highly acidic solutions generally

promote cracking and highly basic solutions generally retard cracking and usually there is little effect of pH on K_{Iscc} over the range of 3 to 10. Carter,[4,48] in reviewing the effects of pH on K_{Iscc} in water and aqueous chlorides, gives the insensitive range as pH 2 to 9. For notched specimens of 300-grade maraging steels Carter[4] lists this range as 0.5 to 11. For smooth specimens of the same steels there is an effect over this range which is associated with pitting. For aqueous sulfides the susceptibility to SCC is affected by pH over the entire acidic range, and the effect is further modified by the H_2S content.[48]

Impressed voltages appear to have little or no effect on K_{Iscc} (at least for low- and medium-alloy steels) but they have large effects on t_f and CGR,[4-6,43,53,157] especially in acidic environments.[4] Minimum in CGR usually occurs under slightly cathodic polarization potentials relative to the freely corroding potential[4-6] with the CGR increasing rapidly with further increase in negative potential. The accelerated crack growth under even moderate cathodic potentials attests to the role of hydrogen cracking in SCC. Where anodic dissolution predominates, exposure below K_{Iscc} can lead to crack blunting and therefore greatly increase crack initiation time. With insufficient test time this may lead to an apparent increase in K_{Iscc}.[167] A large drop in K_{Iscc}, from 103 to 24 ksi·in.$^{1/2}$, was reported for an 18Ni-200 grade maraging steel when cathodically polarized in a 3-1/2% NaCl solution.[168] Maximum test periods were < 1000 h. Dautovich and Floreen in a review paper[157] on maraging steels also report similar examples of a large drop in K_{Iscc} for several different σ_y levels in the 200 and 250 grades. Thus, it may be that the effect of polarization on K_{Iscc} is different for maraging steels than it is for the low- and medium-alloy steels. Alternatively, the maraging steels may reflect insufficient exposure time under freely corroding potentials coupled with an increase in CGR under cathodic polarization. Novak reports that even 10,000 hours, based on his studies, may not be adequate for 250-grade maraging steel exposed to salt water.[177] In fact, the subject of adequate exposure time has received too little attention. Extrapolation of currently accepted K_{Iscc} data largely based on tests usually ranging from 100 h to about 1000 h may lead to erroneous conclusions of maximum allowable stresses for long exposure times of many years.

Kerns, et al.[6] and Carter and Hyatt[48] discuss the role of cathodic poisons in terms of group V and VI elements (P, As, Sb, S, Se, Te), hydride species, CN^-, and sulfur bearing compounds on restricting the recombination of hydrogen atoms to form H_2 gas and in promoting hydrogen entry. The effect of these poisons is to increase the CGR and possibly decrease, but to a much lesser extent, K_{Iscc}.[4-6,53]

As a final comment, on evaluating the integrity of bolting materials by the use of lower-bound K_{Iscc} curves, the many variables affecting K_{Iscc} must be included in any such analysis. Both material variables and environmental variables must be considered. Furthermore, the effects of any of these variables on CGR should be considered if stress transients occur that could result in exceeding the lower-bound K_{Iscc} values.

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5. SUMMARY

The results of an analysis of an extensive literature survey on stress corrosion cracking (SCC) are presented in the form K_{Iscc} versus yield strength (σ_y) plots. The data are for a variety of steels typical of bolting materials. Environments include water, aqueous chlorides, aqueous sulfides, and H_2S and H_2 gases.

The plots of the reported data exhibit considerable scatter. This could be attributed to either material, environmental, or test variables and these possibilities are discussed. Variables such as grain size, composition, or polarization potential may affect K_{Iscc} and crack-growth rate (CGR) or time to failure (t_f) in dissimilar or even opposing ways. It is therefore suggested that an evaluation based on K_{Iscc} should include CGR and/or t_f where possibilities exist of exceeding K_{Iscc}. Uncertainties in published K_{Iscc} data based on too short exposure periods are also emphasized.

Lower-bound K_{Iscc} curves for the plain-carbon and low-alloy steels exposed to water and aqueous chloride environments are consistent with the proposed NRC lower-bound curve, while the corresponding curve for 18Ni-maraging steels falls below the curve proposed by the NRC, especially at the higher strength levels. For the precipitation-hardening stainless steels, at the lower strength levels the curves fall below the lower-bound curve suggested for the maraging steels but merge with the latter curve at stress levels above about 220 ksi σ_y. At high σ_y values it appears that K_{Iscc} becomes insensitive to variations in σ_y for all heat-treatable high-strength steels. The stress value at which this occurs is about 190 and 240 ksi for the low-alloy and high-alloy steels, respectively, with the lower-bound K_{Iscc} apparently levelling off at ~10 ksi·in.$^{1/2}$ for all the steels examined.

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An extensive literature survey on stress-corrosion cracking of a variety of steels was made in response to the need by the U.S. Nuclear Regulatory Commission (NRC) to establish lower-bound K_{Iscc} values for bolting type materials. Materials evaluated include the heat-treatable plain-carbon and Cr-Mo and Cr-Mo-Ni low alloy steels, 17-4 PH and Custom 455 precipitation-hardening stainless steels, and the 18 Ni-maraging steels. An exhaustive survey was made for water, aqueous chloride, and aqueous sulfide environments. We also report limited data on H_2S and H_2 gases, aqueous H_3BO_4, and atmospheric environments. The data are presented in the form of K_{Iscc} versus yield strength. The proposed NRC lower-bound K_{Iscc} curve for the low-alloy steels is consistent with the data reported for the various aqueous environments, but excluding the sulfides. The corresponding lower-bound curve based on reported data for 18 Ni-maraging steels falls below that proposed by the NRC, especially at the high strength levels. The lower-bound curves for the precipitation-hardening stainless steels fall below the lower-bound curve for the maraging steels at the lower strength levels, but they merge at a yield strength of approximately 220 ksi. Reference is also made to crack-growth rate (CGR) and time to failure (t_f). These two parameters frequently were found to vary differently from the corresponding K_{Iscc} values when examined as a function of material or environmental variables. The influence of various material and environmental factors on K_{Iscc}, CGR, and t_f are discussed.

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